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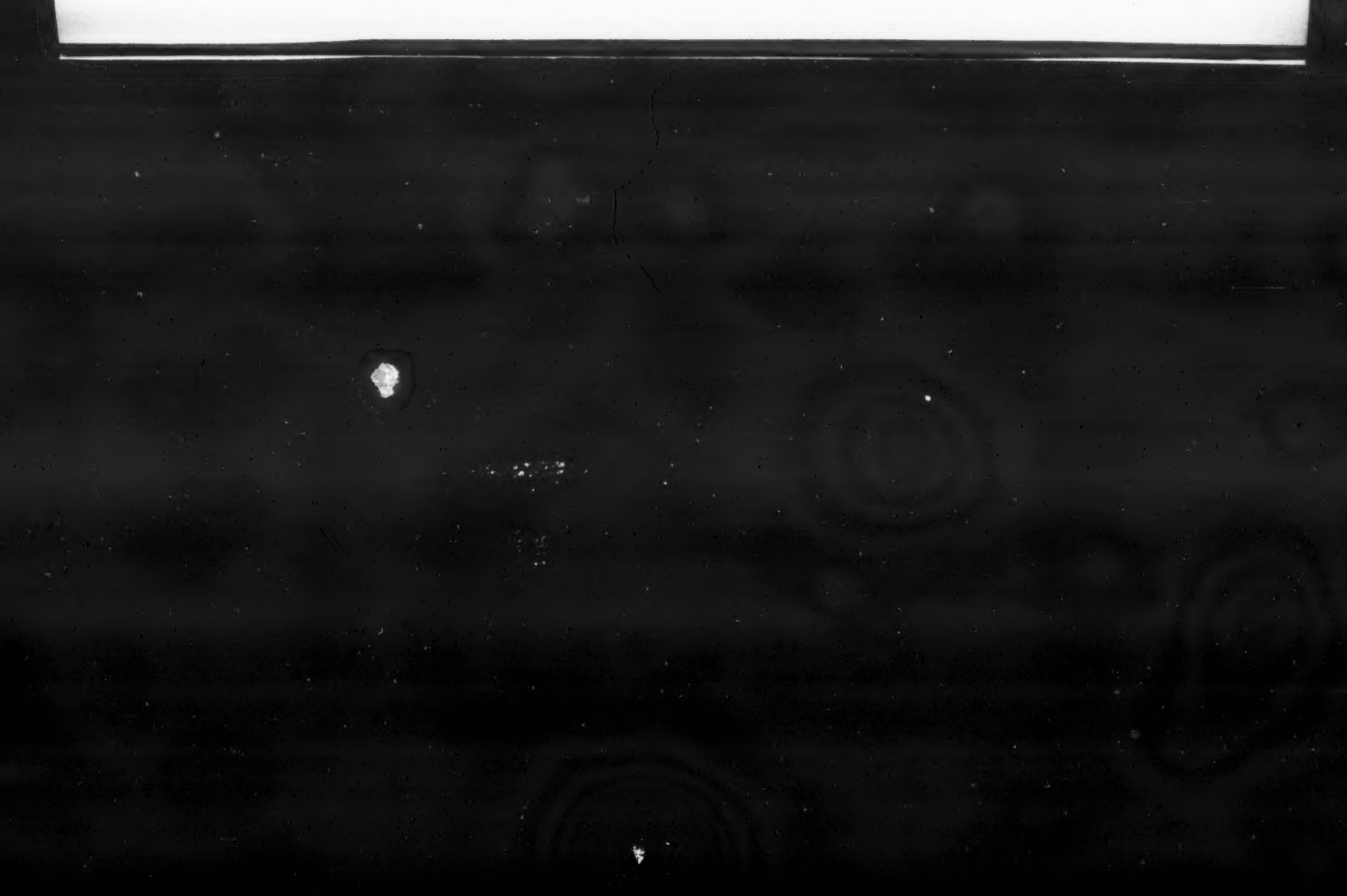


THE

AMERICAN JOURNAL OF SCIENCE

[THIRD SERIES.]

ART. XI.—*On the Relations between the Surface Tensions of Liquids and their Chemical Constitution;* by C. E. LINEBARGER.

THE mathematical and physical parts of the subject of the surface tension of liquids have received much attention, and may be considered as having reached a high stage of development. The basis of the theory was laid by Young,* who regarded a liquid as bounded by a superficial film, behaving like a stretched membrane, and who showed that the form of the surface could be accounted for by taking into consideration the conflict between the surface tension and the other forces acting upon the liquid. This theory was elaborately worked out by Laplace,† Poisson,‡ Gauss,§ Hagen‖ and many others. Numerous investigators have occupied themselves with the experimental verification of the theory and treatment and development of certain physical and mathematical questions pertaining to it.¶ The bibliography relative to the phenomena of capillarity is very extensive; indeed, but few branches of physics have received more attention.

The chemical side of the question, on the other hand, has

* An Essay on the Cohesion of Fluids, Phil. Transactions for 1805; and Young's Collected Works by Peacock, vol. i, p. 418.

† Sur l'action capillaire, Supplément au X livre du Traité de Mécanique Céleste. (1845), Paris.

‡ Nouvelle theorie del'action capillaire (1831), Paris.

§ Principia generalia theoriae figurae fluidorum in statu aequilibrii, Göttingen.

‖ Ueber die Oberfläche der Flüssigkeiten, Berlin Acad. (1845).

¶ For a historical sketch of the subject, see Quincke, Pogg. Ann., cv, I, and Maxwell, in Encyclopedia Britannica.

received comparatively little attention, although it is universally recognized that there must be an intimate connection between the capillarity and chemical constitution of liquids. Mendeléeff* seems to have been the first to have entered this field of investigation, being followed a few years later by Wilhemy;† the work of Quincke‡ on the surface tension of substances at their point of liquefaction should also be mentioned here. Robert Schiff,§ too, has published extensive and valuable facts and deduced therefrom some important conclusions in this domain of research. I will not enter now into a review of these investigations, as they will be frequently referred to in discussing my results.

Three principal methods have been employed in the determination of the surface tension of liquids; 1°, the *capillary tube method*, consisting in observing the height to which a liquid rises in a capillary tube of known bore; 2°, the *drop or bubble method*, consisting in the determination of the shape, and size of drops or bubbles formed in various circumstances: 3°—the method consisting in ascertaining the force necessary to pull a disk of known area from the surface of a liquid. These methods have, indeed, been very much modified in individual cases, but, in general, all may be referred to one or another of the foregoing. The results obtained by different methods have been found to be quite concordant.

Most of the determinations of surface tension have been made with *one* liquid in contact with air, the surface tension of which being so small, could be safely neglected. About the only investigations of the surface tensions of two liquids in contact are those of Quincke (who determined the shape of drops of one liquid resting in another, and the change in the height of a liquid in a capillary tube when subjected to the influence of another liquid, etc.) and of Guthrie.‖ These researches are entirely physical, chemical questions being hardly touched, yet as it was Guthrie's work which induced me to take up this subject, and as his methods resemble mine closely, I will describe some of his experiments and cite some of his conclusions.

From a glass ball, suspended in a funnel-shaped vessel, water was made to drop, the drops being caught in a graduated tube placed at the bottom of the vessel. Funnel and tube were filled successively with air, "turpentol," and benzene, and the flow of water so regulated that a drop fell every five seconds. It was found that there were required to fill the tube up to a

* Compt. Rend., l, 52 and li, 97 (1860). † Pogg. Ann., cxxii. 55, (1864.)
‡ Pogg. Ann., cxxxv. 621. (1868) and *ib.* cxxxviii. 141. (1869.)
§ Liebig's Ann., ccxxiii, 47, (1884) and Gazz. chim. ital. xiv, 137, (1884.)
‖ Proc. Roy. Soc., xiii, 444 and xiv, 22.

fixed mark, 57 drops in air, 27 in "turpentol" and 7 in benzene. As the benzene and "turpentol" had nearly the same specific gravity (see Table I), it is impossible that the difference in the size of the drops should be due to this alone. Guthrie also determined the weight of drops of mercury in various media. I give his principal results in Table I.

TABLE I.

	Specific gravity.	Weight of drop of mercury in	Weight of drop of mercury in air.
Air	0·	0·7654 gram.	0·7654 gram.
Water	1·	0·6462 "	0·6975 "
Glycerine	1·245	0·5579 "	0·6151 "
Benzene	0·864	0·5601 "	0·5982 "
"Turpentol"	0·863	0·4071 "	0·4350 "

Here also, a great difference is observed in the weights of the drops of mercury in benzene and "turpentol;" and, although there is not much difference in the drop-size of glycerine and benzene, yet their specific gravities are far from being the same. Guthrie states as one of the principal results of his work that "the drop size of a liquid, which drops under like conditions through various media, does not depend wholly upon the density of the medium and consequent variation in the weight of the dropping liquid."

Evidently this behavior is due to the chemical constitution of the liquids examined, and the question arises, would not the determination of the surface tension between many other liquids afford valuable data in regard to their chemical constitution? The object of the present paper is to begin the answer to the question. It will require much work in order to find out what laws underlie these phenomena, and this article will contain but a description of the apparatus used, together with some indications of the direction that succeeding investigations should take. The results obtained with the liquids examined show that some importance may be attached to the thorough investigation of the subject.

Experiments of this kind should be carrried out with liquids which are totally or nearly insoluble in one another, but it is difficult, if not impossible, to find liquids fulfilling those conditions. All liquids, which have a perceptible vapor tension at the temperature at which a determination of solubility would be made, must dissolve one another to some extent; for as, in general, every liquid has the power of absorbing to some degree every gas or vapor, one liquid in this case would dissolve the vapor of the other, and it is impossible to distinguish between a solution of a substance in gaseous form and its solution in liquid form. This is shown by the fact that, if water be

shaken up with an ethereal oil, although the liquids are regarded as insoluble in each other, yet the latter imparts its odor to the former. There are, however, numbers of pairs of liquids which are practically insoluble in each other; at least, we are unable by our analytical methods to detect the presence of one in the other—and it is the surface tension of these liquids that it is my purpose to investigate.

At the surface of two absolutely insoluble liquids, there would reign complete repulsion between the molecules; no molecule of one liquid would pass beyond a definite surface concentric with the surfaces of the liquids, and this surface would form an impenetrable barrier to molecules of each liquid. But in the case of liquids which dissolve one another a little, complete repulsion between the molecules prevails no longer, but on the contrary a slight attraction, so that now a molecule of one liquid may pass into the other. This would continue until the "tension" of solution of each liquid was reached. The question arises then, whether, in searching for stoichiometrical relations, the superficial tensions of liquids, saturated with each other, or pure, be employed. Leaving the discussion of the question for the present, we will in this paper make use of those superficial tensions determined with liquids uncontaminated with one another.

It is necessary in work of this kind to select one liquid as a standard of comparison, absolute measurements being less reliable than comparative. Mercury, as regards insolubility, is admirably adapted to the end in view, but I have not found it advantageous to employ it in measurements made by the method I adopted. I have therefore chosen water, the universal standard, as it is insoluble in a sufficient number of organic compounds to receive quite an extended use.

The method consisted in determining:—1°, the number of drops into which a certain volume of water divided in "dropping down" through liquids lighter than water and in "dropping up" through liquids heavier than water; 2°, the number of drops of a liquid "dropping down," if heavier than water, and "dropping up" if lighter, through that standard.*

The bulb of a small pipette was so shaped and ground that it fitted snugly (like a stopper in a bottle) in the neck of a cylindrical vessel, provided at its lower end with a stop cock. An inch above the bulb, a second much smaller bulb was blown, and between it and the larger bulb as well as an inch or so below the latter, marks were scratched. The lower stem of the pipette was bent out so as to run down close to the side of the cylindrical vessel nearly to the bottom, where it was turned

* For an extension of the meaning of "drop," I refer to Guthrie's work (loc. cit.)

up through two right angles, the orifice being exactly in the axis of the cylinder, no matter how the pipette was inserted. It is of some importance that the orifice be always placed in such a position, that the drops may be discharged vertically. A second pipette with a straight stem, so as to discharge bubbles downwards, was also employed. To make a determination, the pipette (the curved-stemmed one, if the dropping liquid is the lighter, and the straight-stemmed one, if the dropping liquid is the heavier) is filled with one of the liquids by suction through a long rubber tube slipped over the upper end of the pipette and provided with a pinch cock. It is then inserted in the cylinder which has previously been filled with enough of the other liquid to stand an inch or so above the orifice of the pipette. The free end of the rubber tubing is slipped over a piece of glass tubing, so shaped that it can be held conveniently in the hand and closed with the thumb. The thumb is now kept tightly pressed over the tube, and the pinchcock opened. A few drops fall rapidly at first, before the pressure becomes sufficiently reduced. By lifting the thumb, air may be admitted and the liquid made to drop at any desired rate. After a little practice, the raising of the thumb and the counting of the drops are done almost instinctively. If the time of the growth of the drops is to be noted, a watch is placed at the side of the apparatus, and the falling of the drops made to coincide with the movements of the second-hand. This is very easily done. Suppose, for instance, one wants the drops to form at the rate of one a second. Then, by beginning to count when the second-hand crosses a quarter-minute mark, fifteen drops should have fallen when the second-hand passes the next quarter-minute mark, thirty drops, when it passes the next, and so on. In this way, with very little effort, a strict account of the time taken by the drops in forming can be kept. The number of drops, into which the volume comprised between the two marks divided was taken in every case. If the last drop before the mark was reached did not leave the residual liquid nearly flush with the mark, which was generally the case, another drop was allowed to form, and, by observing the volume it occupied in the stem of the pipette, an estimation to a quarter of a drop could be made, which was added to the number of drops counted. When one determination had been made, the pipette was removed, wiped free from the outside liquid, refilled and again inserted in the cylinder. The liquids in the outside vessel are easily drawn off separately by means of the stop-cock.

In regard to the accuracy of results obtained by this apparatus, it was found that determinations of drops forming at the same rate and dropping upwards, seldom differed, if the

liquids were quite insoluble in each other, by more than one drop in a hundred. If, however, the liquids were somewhat soluble in each other, quite variable results would be obtained until the one liquid was saturated with the other. Thus water in dropping up through nitrobenzene which was not changed in three determinations gave for the first series: 1°, 136 drops—2°, 142 drops and 3°, 155 drops; and for the second 1°, 133 drops—2°, 146 drops and 3°, 161 drops. The first two determinations agree fairly well; they were made with water dropping through dry nitrobenzene. But as the nitrobenzene became more and more contaminated with water, the surface tension of the liquids changed as shown above in the number of drops. Three independent determinations of the number of drops of water saturated with nitrobenzene up through nitrobenzene saturated with water gave—1°, 112 drops—2°, 110·5 drops—3°, 111 drops,—which are entirely concordant. This slight solubility of liquids in one another complicates matters greatly, and renders stoichiometrical conclusions based upon results obtained by measurements of superficial tensions of liquids by the above method somewhat unreliable. Without doubt, however, by a further study of these phenomena, we shall learn how to make due allowance for such disturbing influences. Of the liquids which I have examined, not one, with the exception of nitrobenzene and bromoform, has given any too variable results.

The time of the growth of drops has a little influence upon their size. For an elaborate investigation of the influence of time upon drop-size I must refer to Guthrie's papers. In my experiments, it was impossible to choose a certain fixed rate at which all drops should form, since a large drop requires a longer time for formation than a small one; hence I made the drops form at such a rate as seemed necessary for the attainment of the maximum size.

In the case of liquids heavier than water, falling through water, the orifice of the pipette was wetted by the water, and hence the drops formed upon the inner circumference of the orifice, which, however, in my instrument differed very slightly from that of the outer. It was difficult to obtain concordant results in this case, since the drops could not be prevented from falling by "twos" and "threes." That is, a drop would commence to form slowly, when, all of a sudden, it would increase rapidly in size, fall off and be followed by another, which would, however, take a longer time for formation. There seems to be a sort of pull exercised by the first drop on the second. Sometimes two drops would issue rapidly from the orifice to be followed more slowly by a third. This, rendering the rate of formation irregular, caused the results to

be somewhat variable, since the first drop was evidently smaller than the succeeding ones. Not so much confidence, then, is to be placed in the results obtained by the dropping of heavier liquids through lighter, as of lighter up through heavier ones, since in the latter case no such phenomena were observed.

In the dropping of the liquids, besides the main drop, a much smaller secondary drop would also form almost invariably. In general, the larger the main drops, the smaller the secondary drops, so that but a slight error is introduced in neglecting the latter, when the former are comparatively large. The formation of these secondary drops may be explained as follows: The main drop, just before breaking loose, is connected to the mass of the liquid by a short cylinder, the circumference of which is that of the orifice of the pipette. When the large drop tears itself from the connecting cylinder, a certain impulse or shock is communicated to the latter, so that it is severed from the mass of liquid, and under the action of superficial tension and gravitation assumes a spheroidal form, appearing as a drop. In this paper, I have not attempted to make any corrections for these secondary drops, although, without doubt, their appearance is intimately connected with the superficial tension of the liquid in question.

When water was dropped down through other liquids, it would run up around the orifice of the pipette so that the drops were larger than they should be, and consequently the results were quite variable. This was prevented by the following simple device. A test tube just small enough to enter the outer vessel of the dropping apparatus was cut off so as to form a tube open at both ends about two inches long. In one end was fitted a cork through which a hole was pierced so that the stem of the pipette could be thrust through it. In the other end was set a thin ring of cork made by cutting a cross-section of a good soft cork and filing it out. This ring was cut into four times so as to form four shallow clefts situated 90° apart. In these clefts were inserted fine-spun threads of glass, so that they crossed at the center of the ring. The orifice of the pipette was shoved down close enough to the intersection of the glass threads that a drop, when nearly half formed would touch it. The drop then, instead of running up the sides of the pipette would be confined to the orifice, the crossed threads serving as a support for the drop during its formation. With this little device, the determinations were entirely concordant.

We will first take up the discussion of the results obtained with liquids lighter than water. These were all hydrocarbons of the benzene series from the establishment of Kahlbaum

labelled chemically pure. I made no special tests for their purity, as for my purpose they might be considered pure enough.

The results are given in Table I. In the first column are given the names, in the second the formulæ of the substances, while in the third are recorded their densities at 20° as found in the literature. The fourth column contains the weights in grams of drops of the respective hydrocarbons in water, and the fifth the weights of drops of water in the hydrocarbons.

I have not calculated the surface tension, as I did not know the radius of the orifice of the pipette; as it deals here of relative rather than absolute values, that is of little moment.

TABLE II.

I.	II.	III.	IV.	V.
Benzene	C_6H_6	0·879	0·0405	0·1932
Toluene	$C_6H_5 . CH_3$	0·865	0·0403	0·1920
o-xylene	$C_6H_4 . (CH_3)_2 1·2$	0·75	0·0405	0·2010
m-xylene	$C_6H_4(CH_3)_2 · 1·3$	0·87	0·0406	0·1910
p-xylene	$C_6H_4(CH_3)_2 · 1·4$	0·862	0·0318	0·1579
Cumene	$C_6H_5 · CH(CH_3)_2$	0·87	0·0262	0·1263
Pseudocumene	$C_6H_3 . (CH_3)_3 . 1·3·4$	0·86	0·0314	0·1536
Ethylbenzene	$C_6H_5 . C_2H_5$	0·866	0·0178	0·0826
Mesitylene	$C_6H_3 . (CH_3)_3 . 1·3·5$		0·0141	0·0725

We notice first that the determinations of the size of the drops of the hydrocarbons in water and of water in the hydrocarbons give relatively the same results. If the numbers in the fourth column be multiplied by *five*, the numbers in the fifth column are approximately obtained. This shows that the two methods give strictly comparable results.

The weights of the drops of benzene, toluene, *o*-xylene and *m*-xylene in water, as well as the weights of water-drops in them, are the same, if due allowance be made for unavoidable errors of observation. The superficial tensions of these organic liquids in contact with water have then equal values. In passing to *p*-xylene, there is a considerable decrease in the weights of the drops. This is evidently due to the *para*-position of one of the methyl groups, for pseudocumene, which contains a *meta*- as well as *para*-methyl group, has the same drop-size as *p*-xylene. While the introduction of a *meta*-methyl group in benzene derivatives seems without influence upon the superficial tension, the introduction of a *para*-methyl group exercises, on the contrary, a decided influence. Yet three *meta*-methyl groups, as in mesitylene, cause the surface tension to decrease greatly. Again it is to be observed how different are the drop-sizes in the case of toluene or methyl-benzene and ethyl-benzene. Cumene or isopropylbenzene also has a sur-

face tension greater than that of ethylbenzene, but less than that of toluene.

The laws which underlie these phenomena can be discovered only when the data are much more numerous, although with the above liquids important hints are given as to how those laws will be stated. We find further that the specific gravities of these hydrocarbons have but little, if any, influence upon their surface tensions. True, the differences of density are slight, but it is seen that benzene with the greatest and *o*-xylene with the least density have the same drop-size. And again, the weights of the drops of toluene and ethylbenzene, liquids of almost the same specific gravity, are widely different. Guthrie's first law, as stated above, seems to be verified by these facts.

Of the few liquids heavier than water which I examined, the majority have a similar constitution. These were all carefully purified and only portions boiling within a fraction of a degree taken. The results of the determinations are given in Table III., where Column I, gives the name and Col. II, the formula of the liquids. The specific gravities at 0° for the most part are entered in Col. III, and the weights of drops of water in the organic liquids in Col. IV. Cols. V and VI give the weights of the drops of the liquids into water, orifices of different sizes being used.

TABLE III.

I.	II.	III.	IV.	V.	VI.
Nitrobenzene	$C_6H_5 . NO_2$	1·2	0·0518	0·0328	
Carbon disulphide	CS_2	1·29	0·0667	0·0384	0·0461
Amyl bromide	$C_5H_{11} . Br$	1·28	0·0666	0·0382	0·0460
Ethyl bromide	C_2H_5Br	1·47	0·0236	0·0182	0·0221
Benzene bromide	$C_6H_5 . Br$	1·517	0·0283	0·0237	0·0280
Chloroform	$CHCl_3$	1·52	0·0229	0·0278	0·0362
Ethylene bromide	$C_2H_4Br_2$	2·18	0·0106	0·0137	0·0183
Bromoform	$CHBr_3$	2·83	0·0069		0·0176

If the numbers in Cols. V and VI, be plotted as ordinates and the corresponding densities as abscissas, the resulting curves will be seen to be parallel, showing similarity, as might be expected, in the results obtained with different sized orifices.

Regarding Col. II, we see that carbon disulphide and amyl bromide, liquids of the same specific gravity, have the same drop-size, while benzene bromide and chloroform, also liquids of about the same density, have quite different drop-sizes. A difference in the chemical constitution of these last two bodies may explain their difference in surface tension; we see also that, although the density of nitrobenzene is less than that of amyl bromide and carbon disulphide, its drop-size is not greater.

The influence of the specific gravity in the case of liquids heavier than water is much more marked than in the case of those lighter than it. Thus the table shows that with the exception of the benzene derivatives, the drop-sizes diminish quite regularly, as the specific gravities increase. The anomaly in the behavior of the benzene molecule is undoubtedly due to its constitution.

The foregoing work was performed with the purpose of developing and testing methods, rather than that of accumulating data; it is to be regarded as a series of "orientation" experiments, which have shown in what direction it is best to continue. Investigations of this sort will certainly throw a much clearer light upon the nature and workings of the molecular forces, a knowledge of which is of the greatest importance.

Chicago, Ill.

Art. XII.—*The Gold Deposit at Pine Hill, California;* by Waldemar Lindgren.

General Type.—Veins and seams of barite, carrying gold and silver, distributed through a kaolinized zone in diabase and diabase porphyrite.

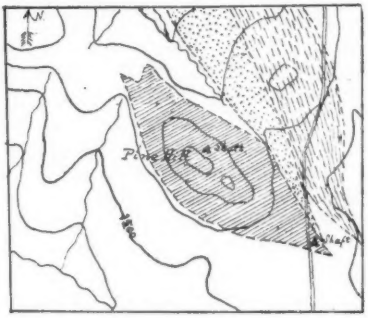
The auriferous deposits of California have, as well known, two principal modes of occurrence: Secondary as gold-bearing gravels and sands of Tertiary or Pleistocene age, and primary as fissure veins, largely of late Mesozoic age. The latter occur in great variety of formations, but on the whole, avoid the large granite areas and seem to prefer the contact of sedimentary slates with eruptive, unaltered, or dynamically metamorphosed masses. The form of the auriferous veins also varies considerably between normal, regular fissure veins and networks of minute irregular fractures.

With all diversity in surroundings and form, the vein material or gangue and the mineral associations are simple and subject only to rare and slight variations. The gangue is nearly always quartz, the mineral association native gold, sulphides of iron, copper, lead, and zinc,—frequently also arsenical pyrites, rarely tellurides. Dolomite and calcite accompany the quartz at certain veins, but even then the quartz is usually the principal ore carrier.*

* The association of native gold with calcite in Shasta County, California, has been noted by Mr. G. F. Becker (Statistics of the Precious Metals, Tenth Census, vol. xiii. p. 24), and from Trinity County, California, by Mr. J. S. Diller (this Journal, vol. xxxix, 1890, p. 160).

The deposit to be described here differs strangely from the common type. Pine Hill is situated about eleven miles south-southwest of Grass Valley, in Nevada County, and a few miles north of Bear River; it is in the lower rolling foothills of the Sierra, and the summit of the little knoll bearing the above name rises to eighteen hundred feet above the level of the sea; from the top an extensive and beautiful view is obtained westward over the Sacramento valley, and eastward toward the crest of the snowy range.

1.



Sketch of Pine Hill and vicinity, Nevada Co., Cal.
Scale 2 inches = 1 mile. Contour Interval 100 feet.

A. Diabase or Porphyrite. B. Decomposed Zone.
C. Serpentine. D. Quartzite and Slate.

Toward the south and west a large area extends occupied by massive diabases and diabase porphyrites in which auriferous veins are but seldom met with; to the east of Pine Hill there is a somewhat complicated area of quartzites and clay slates, serpentines and gabbros sometimes dynamically metamorphosed and containing a few veins of gold quartz, usually very capricious as to the distribution of the metal. A part of this area is shown on the sketch. To the northwest, beyond the limits of the map, there is a large and massive area of granite, diorite and gabbro with very few auriferous deposits.

The diabase and diabase porphyrite which form the prevailing rock of the vicinity, are massive, fine grained and not a little affected by secondary processes giving rise to uralite, chlorite, secondary quartz, etc. The diabase porphyrites appear to prevail along the crest of the ridge, of which Pine

Hill forms the cumulating point. Surrounding the latter there is an area of intense decomposition, somewhat ill defined but approximately one mile long and from one to two thousand feet wide, of a very different character from the ordinary chloritization and uralitization. The rocks within this area are converted to a soft, porous, reddish brown to yellowish mass, from which calcium, magnesium, iron and the alkali metals together with a part of the silica are removed; the ultimate product of this decomposition, which I think should be regarded as having been effected by thermal waters, alkaline in character, is a soft white nearly pure kaolin* or hydrated silicate of aluminium. If there exists a large quantity of this pure white kaolin it could no doubt be utilized for keramic art; the somewhat impure substance would do very well for the manufacture of ordinary pottery.

In many places the zone of decomposed and kaolinized diabase is impregnated with irregular veins and seams of barite or heavy spar, and with this mineral the gold is closely connected. The largest barite mass appears on the northeast side of the hill and about three hundred feet from the crest; a shaft fifty feet deep was sunk here a few years ago and some ore extracted. The deposit not being worked at the time of my visit, the shaft was inaccessible. It did not appear as if a well defined and extensive vein of barite were present, but rather as a local enlargement of one of the numerous smaller irregular barite seams. The ore on the dump was largely composed of barite mixed with limonite and decomposed country rock. No sulphurets, but a few stains of sulphate of copper were noticed. Although no normal vein quartz occurs, there are in various places seams and smaller masses of a fine granular aggregate of quartzitic appearance but doubtless due to secondary processes. Assays were made on a series of specimens, both of ore and country rock, by Dr. W. H. Melville, in the laboratory of the U. S. Geological Survey. Special precautions were taken to insure correct results and the exceptionally pure litharge used contained only 2 cents of silver to the ton.

The assays in general show a large relative amount of silver—more than is usual in the gold quartz veins. The proportion of gold to silver by weight varies from 1 : 1 down to 1 : 5, or in value from 20 : 1 to 4 : 1. The metal resulting from the reduction of this ore would be a doré bullion similar to that yielded by the Bodie (Mono County) quartz veins.

The assay of the ore from the dump, composed of barite

*In a specimen of this white substance the water was determined quantitatively by Dr. E. A. Schneider, who obtained almost exactly the theoretical amount for kaolin, viz: 13·8 per cent H_2O.

and some limonite, gave a paying amount of gold with silver in the proportion of 1:2 by weight; a piece of very pure white barite selected for the purpose, and shown under the microscope to contain no visible gold or sulphurets, gave the same amount of gold, but much more silver, the proportion between the two metals being 1:5. Only a part of the silver exists as an alloy with gold, for a pan of the ore washed out gave a considerable quantity of quite *yellow* gold in very fine particles; hence it may be permitted to surmise that the silver partly occurs as a chloride. A piece of barite with much limonite gave an exceptionally high return with but little silver, the proportion being 1:1.

Near the crest of the hill there are several little prospect holes in the decomposed mass. One specimen shows a brownish to gray compact decomposed rock permeated with little seams of barite. This gave 0·063 oz. gold and 0·163 oz. silver to the ton, or $1.26 in gold and $0·16 in silver, the proportion in weight being about 1:3.

A specimen from the summit of Pine Hill is a soft crumbling mass largely composed of kaolin, pink or yellow in color and showing on fresh-fractured surfaces traces of the grain of the original rock. It was not expected that this mass would yield any results, but it proved to contain 0·0375 oz. gold, and 0·075 oz. silver per ton, or respectively $0.75 in gold and $0.07 in silver. No barite is visible in the rock, but a quantitative determination by Dr. E. A. Schneider showed it to contain 0·34 per cent $BaSO_4$.

At the southeast end of the decomposed area a shaft has been sunk to a depth of about one hundred feet, and traces of gold are reported to have been found.

It is thus reasonably certain that this whole altered mass of diabase and porphyrite is auriferous; that barite in very varying quantities is distributed through it, and that the quantity of the gold contained is approximately proportional to that of the barite, or, in other words, that the barite acts as the carrier of the gold. The primary mineral combination was probably native gold, pyrite, rich silver ores, and barite.

Whether the kaolinized zone is connected with large fissures, it is not possible to say from the present slight underground developments; on the surface, at least, there are no indications of such a connection, yet a channel must have existed for the passage of the waters effecting this far-reaching alteration of the rocks.

In the foothills of Yuba County and also in massive diabase there is a zone of extreme decomposition resembling somewhat the one here described, but associated with a considerable amount of secondary, fine grained, quartzose and chalcedonic

rocks. The larger part of this area carries minute quantities of gold and silver, the latter predominating, the ratio being 1 : 6 by weight.

Native gold is but seldom associated with barite, and only, as far as I am aware, in silver-gold deposits in which the former metal predominates. Barite is a rare mineral in California, and does not occur in the normal gold quartz veins. Native gold with barite is mentioned as a rare occurrence from southern Colorado,* and Mr. Hanks, in his description of California minerals,† refers to a specimen of barite with gold from the Malakoff hydraulic mine, near North Bloomfield, Nevada County. It may be stated in this connection that there is a deposit of barite in clay slates seven miles east-northeast of North Bloomfield and cropping out on the North Bloomfield ditch. I am not aware, however, that it contains any gold.

Another instance of gold connected with barite is found in Yuba County at the junction of the North and Middle Yuba Rivers. A streak of chloritic schist, about twenty feet wide, is here impregnated with pyrite and chalcopyrite and traversed by seams of calcite and barite carrying the same minerals. The brown surface croppings carry native gold, but it is probably all derived from the decomposition of the pyrite.

U. S. Geological Survey, Washington, D. C., April, 1892.

Art. XIII.—*A new occurrence of Ptilolite;* by Whitman Cross and L. G. Eakins.

In this Journal for August, 1886, we described the first observed occurrence of a new hydrous silicate of alumina, lime and alkalies, to which we gave the name *ptilolite.* The mineral was remarkable for its high percentage of silica, the analysis leading to the formula $R'Al_2Si_{10}O_{24} . 5H_2O$. Inasmuch as the polysilicic acid of this formula was elsewhere unknown, Prof. P. Groth‡ expressed the belief that the material analyzed must have been contaminated with chalcedony, upon which the ptilolite was deposited. While it has seemed to us that the care exercised and the method used in the purification of the original ptilolite material analyzed must exclude the possibility of such contamination, we are glad to be able to announce a second occurrence of this interesting mineral the investiga-

* See the interesting paper by Mr. R. C. Hills, "Ore Deposits of Summit District, Colorado," Proc. Col. Scientific Soc., vol. i, p. 24.

† Fourth Annual Report California State Mining Bureau, p. 78.

‡ Tabellarische Uebersicht der Mineralien, 2d ed., p. 149, 1889.

tion of which fully confirms the earlier analysis. The work of L. V. Pirsson* upon mordenite has, however, fully confirmed the existence of silicates with the ratio between silica and bases which was found in the original ptilolite.

The new occurrence of ptilolite is in Custer County, Colorado, about three miles southeast of the mining town of Silver Cliff. The rock containing the ptilolite occupies but a few square feet of surface on a low ridge of rhyolitic breccia reaching out from the volcanic center of the Rosita Hills,† and we are indebted to Mr. Thomas Charlton of West Cliffe for calling our attention to the peculiar and seemingly insignificant occurrence. The rock is a dull green felsitic mass containing many nearly round vesicles most of which are less than 5^{mm} in diameter while a few are somewhat larger. They occupy about one-third of the rock. The dull green mass proves on microscopical examination to be a completely devitrified pitchstone, and it is probable that this vesicular rock is a remnant of the upper portion of a rhyolite flow seen at a lower level near by, though the connection is covered by "wash" and soil. The rock contains few phenocrysts of feldspar, but a fluidal structure curving about the vesicles is distinct. Devitrification proceeded from pearlitic cracks.

The vesicles of the rock have very smooth walls and most of them have a thin crumbling lining of a pale yellowish or white substance. A few cavities contain a shell of pale bluish glistening quartz crystals and some are entirely filled by this mineral. More abundant than the quartz is a colorless or pale blue mineral in minute thin tablets which are often grouped in sheaves or bundles. This mineral was identified as barite by chemical analysis.

Nearly all the vesicles contain a white mineral in extremely minute needles which form a loose, felt-like mass. Usually they fill the cavity and with a curved dissecting needle one can often remove a little white ball of the substance, apparently perfectly pure. The strong resemblance of this mineral to the ptilolite of Green Mountain was evident at first glance.

Microscopical study of the needles shows that they are transparent, colorless, seldom more than ·005^{mm} and often less than ·001^{mm} in thickness. Only when a number of the needles are arranged in parallel position in a bundle do they perceptibly affect polarized light. In such cases they extinguish ± to the length axis, and by the aid of the quartz plate a negative optical character can be made out.

* On Mordenite, this Journal, vol. xl, p. 232, 1890.

† The geology of this region will be described in a forthcoming monograph of the U. S. Geological Survey. A geological sketch of the Rosita Hills, by Whitman Cross, was published in the Proceedings of the Colorado Scientific Society for 1890, p. 269.

The obtaining of material for chemical analysis was in this case even more difficult than with the original ptilolite. About six cubic feet of the rock was broken up and the tufts and balls of the downy mineral removed with a curved needle. After several days work with an assistant an amount was obtained which on final purification gave a trifle over half a gram of pure material. This final purification, as in the case of the original ptilolite, was made by stirring the mineral in water, breaking up the felt-like aggregates as far as possible, allowing the heavier portion to settle, and pouring off; this operation being repeated upon the residue until most of the ptilolite was washed out. The collected washings were allowed to settle over night, the bulk of the water syphoned off, and the stirring and pouring off repeated until a product was obtained in which no gritty substance whatever could be detected on careful testing with the rounded end of a glass rod. The material so obtained, after drying on the water bath, was left loosely covered, exposed to the air for several days. Dried for eight days over sulphuric acid it lost 3·84 per cent, all of which was regained in twenty-four hours in the air, and in this condition it was analyzed. Like the original mineral this new ptilolite is but very slightly attacked by hydrochloric acid, and the prolonged action of hot concentrated sulphuric acid is required for complete decomposition.

Analysis and ratios (Analyst, L. G. Eakins):

SiO_2	67·83		1·131	10·1
Al_2O_3	11·44		·112	1·0
CaO	3·30	·059		
K_2O	0·64	·007	·109	·98
Na_2O	2·63	·043		
H_2O	13·44		·747	6·67
	99·28			

The water was fractionated as follows:

At 110°	2·62	·301	1·79
125°	1·31	·073	·65
300°	5·41	·301	2·69
red heat	3·10	·172	1·54
	13·44		
Loss over H_2SO_4	3·84	·213	·190

For comparison with the above, the analyses and ratios of the original ptilolite and of mordenite are appended:

Ptilolite, Green Mt, Colo.			Mordenite (L. V. Pirsson)		
SiO_2	70·35	10·06	SiO_2	66·40	9·88
Al_2O_3	11·90	1·00	Al_2O_3	11·17	1·00
			Fe_2O_3	0·57	
CaO	3·87		CaO	1·94	
K_2O	2·83	·96	MgO	0·17	1·01
Na_2O	0·77		K_2O	3·58	
			Na_2O	2·27	
H_2O	10·18	4·86	H_2O	13·31	6·60
	99·90			99·41	

The empirical formula derived from the analysis of the new ptilolite is: $R'Al_2Si_{10}O_{24}+6\frac{2}{3}H_2O$, which it will be noticed is the same as that of mordenite, but as Pirsson has already pointed out (loc. cit.), the two minerals are physically very unlike.

The two ptilolites themselves are entirely similar both in their manner of occurrence and in their physical properties. In chemical composition the only difference is that the new mineral contains 3·26 per cent more water than the old, with a proportional reduction of the other constituents; the molecular proportions of silica and bases being the same in both occurrences.

This excess of water may possibly be accounted for by the different conditions under which the two minerals were analyzed. The original analysis was made in Denver, the present one in Washington, that is, one in a very dry, the other in a very damp climate. Both minerals were air dried, and as both evidently hold a considerable part of the water very loosely, it seems not unreasonable to suggest that the dry Denver atmosphere may have had sufficient desiccating power to remove part of the water from the mineral analyzed there. In fact, on account of this atmospheric difference, water determinations in the two minerals, especially at low and medium temperatures would hardly be directly comparable, since it is now recognized that at a given temperature the relative humidity of the surrounding atmosphere may markedly influence not merely the rate, but also the total amount, of loss on drying.

The fractional determinations in the new ptilolite show that 3·10 per cent of the water is stable at 300° C., so that this may be basic. In the original material no such line of demarkation in the water was detected, although it was thought then that sufficient care was used, but one important factor was not then so well recognized as it is now, namely, the element of time in drying minerals of this type, and it may be that time enough was not given at each temperature before increasing it. The

fact, however, that a comparatively high temperature was found necessary for the total expulsion of the water, would render it easy to admit the possible basicity of part of it in case other considerations made such an admission advisable in order to construct a rational formula for the mineral.

In mordenite no attempt seems to have been made to fractionate the water, except to determine the loss (3·6 per cent) at 100°, so here we have no evidence as to whether it may be basic in part or not.

The fact that in the new ptilolite we have direct evidence that a definite portion of the water may be basic, and that no good ground against such an assumption exists in the analysis of the original mineral, makes it necessary to carefully consider this point in any attempt to theorize upon the structure of the ptilolite molecule. Prof. Clarke has kindly written a note on the theoretical formula of ptilolite and of the allied mordenite, which appears in the succeeding pages.

In view of their similar mode of occurrence, of their identical physical properties, of their exact chemical agreement except as to water, and of the considerations concerning the water presented above, there can be no question but that these two downy minerals belong to one and the same species—ptilolite.

In comparing the analyses of mordenite and the two ptilolites a great variation in the amounts of lime, soda and potash is noticeable. In mordenite the molecular ratio is $CaO : K_2O : Na_2O = 1 : 1 : 1$; but no simple ratio exists in ptilolite.

The Green Mountain mineral is relatively poor in soda, the Silver Cliff mineral in potash, and the mordenite in lime, facts which seem very strange when we consider the rocks in which the minerals occur. Mordenite occurs in a basalt, but is richest in potash and poorest in lime; the original ptilolite occurs in an andesite of medium composition and is poorest in soda; and the new mineral occurs in a devitrified pitchstone and is poorest in potash. The last instance is most remarkable, for the pitchstones of the region about are usually rich in potash and contain but very small amounts of lime. Analysis of the greenish felsitic mass in which the ptilolite occurs shows, however, that the entire rock has in this case undergone a decomposition which brings it to a constitution similar to that of ptilolite. The lime, at least, of this ptilolite was not derived from the surrounding rock, unless secondarily. The analysis of the rock containing the ptilolite of Silver Cliff is as follows (Analyst, L. G. Eakins):

SiO_2	65·67
Al_2O_3	13·48
Fe_2O_3	1·51
MnO	tr.
BaO	·32
CaO	2·41
MgO	·31
K_2O	2·42
Na_2O	1·52
SO_3	·28
P_2O_5	tr.
H_2O	12·27
	100·19

APPENDIX.—*Note on the Constitution of Ptilolite and Mordenite;* by F. W. CLARKE.

THE data given in the foregoing paper by Cross and Eakins, especially as regards the dehydration of ptilolite, lead to quite simple formulæ for the three minerals under discussion. The moment that we recognize the fact that part of the water in each mineral may be basic, the apparent anomalies disappear; and the compounds reduce at once to similar salts of the acid $H_2Si_2O_5$, which acid is well known in the species petalite and milarite. Both ptilolite and mordenite become simply representable by the general formula

$$Al_2(Si_2O_5)_5R'_4 + n\text{Aq}.$$

the only serious uncertainties being in respect to the hydration.

In brief, the following salts appear to be represented in the three minerals, commingled in simple ratios:

1. $Al_2(Si_2O_5)_5CaH_2$. 3 aq.
2. $Al_2(Si_2O_5)_5CaH_2$. 6 aq.
3. $Al_2(Si_2O_5)_5K_2H_2$. 6 aq.
4. $Al_2(Si_2O_5)_5Na_2H_2$. 6 aq.

Three of these are entirely alike in type:- but the first, containing only half the normal quantity of crystalline water, is assumed in order to account for the low hydration of the original, Green Mt. ptilolite. Upon this basis the three minerals investigated may be assigned the following theoretical compositions:

Ptilolite, Green Mt., molecules 1 and 3, in ratio of 3 : 2
" Silver Cliff " 2 " 4, " " 1 : 1
Mordenite, " 2, 3, 4, " " 1 : 1 : 1

Reducing soda to potash in the Green Mt. ptilolite, and potash to its equivalent of soda in the Silver Cliff minerals, and recalculating to 100 per cent, we get a fair comparison between the actual analyses and theory.

	Ptilolite.			
	Green Mt.		Silver Cliff.	
	Found.	Calc.	Found.	Calc.
SiO_2	70·15	69·22	68·45	67·64
Al_2O_3	11·86	11·77	11·55	11·50
CaO	3·86	3·88	3·35	3·16
K_2O	3·98	4·34	----	----
Na_2O	----	----	3·09	3·49
H_2O	10·15	10·79	13·56	14·21
	100·00	100·00	100·00	100·00

In mordenite, reducing Fe_2O_3 to Al_2O_3 and MgO to CaO, the comparison comes out as follows:

	Found.	Calc.
SiO_2	66·88	66·77
Al_2O_3	11·62	11·35
CaO	2·17	2·07
K_2O	3·62	3·49
Na_2O	2·29	2·30
H_2O	13·42	14·02
	100·00	100·00

Considering the scarcity of material, and the difficulties attending its purification, these agreements are in the main satisfactory. In the water determinations, however, there is an uncertainty. In the theoretical composition assigned to the Silver Cliff ptilolite, one seventh of the water, a trifle under two per cent, is basic. But 3·10 per cent were found to be stable at 300°. What weight can be assigned to this discrepancy is not yet determinable, but it can hardly be regarded as an insuperable objection to the proposed formulæ. The general character of the salts, their essential types, seems to be fairly well established.

ART. XIV.—*The separation of Magnesium Chloride from the Chlorides of Sodium and Potassium by means of Amyl Alcohol;* by R. B. RIGGS.

THIS separation depends on the insolubility of the chlorides of sodium and potassium and the solubility of magnesium chloride in amyl alcohol. That the alkaline chlorides are insoluble in this reagent has already been shown by Gooch in his paper* on the separation of sodium and potassium from lithium by the action of amyl alcohol on their chlorides. In the same paper attention is also called to the solubility of anhydrous magnesium chloride. The difficulty, if there be any, lies in dehydrating this chloride. Attempts to dehydrate magnesium chloride by direct heat result in a more or less complete decomposition. This is also found to be the case where the salt, in a solution containing no free acid, is rendered anhydrous by means of boiling amyl alcohol. The amount decomposed, in this latter case, is however relatively small. The decomposition products are the oxide and hydrochloric acid. It is therefore necessary either to introduce conditions which will prevent such decomposition or to find a means of redissolving any oxide that may be formed. The use of hydrochloric acid gas was deemed impracticable. To use the acid solution was regarded as objectionable† because, in so doing, water, the very substance to be driven out, would be brought into the solution. Experiments were made in which, after the water of the magnesium chloride solution had been nearly or quite expelled by boiling with amyl alcohol, a few drops of benzyl chloride were added and the dehydration completed. The salt dissolved without leaving a residue. So little benzyl chloride was used that it did not seem probable that its use could materially affect the solubility of the chlorides of potassium and sodium. Working on this supposition a series of separations were made the results of which are given below.

Standardized solutions of the several salts were used. The solution of magnesium chloride was prepared from the carbonate, which had been twice precipitated from moderately strong solutions of the chloride. The chloride thus made was free from alkalies and wholly soluble in amyl alcohol. The strength of this solution was found by treating weighed por-

* Am. Chem. Jour., vol. ix, p. 33.

† This objection was afterwards found to be groundless.

tions with sulphuric acid in excess, evaporating, igniting and weighing as sulphate.*

The potassium chloride was obtained from the chlorate. The chloride of sodium was prepared from sodium hydrate (obtained from sodium) and further purified by precipitation by hydrochloric acid.

Both solutions were standardized by evaporating weighed portions to dryness, drying at about 300° C. and weighing.

The solutions, thus standardized, were found to contain respectively 0·5293 grm. NaCl, 0·5412 grm. KCl and the equivalent of 0·4146 grm. MgO to the 100 grams.

Three series of separations, containing severally mixtures of magnesium chloride and one or the other or both of the alkaline chlorides, were made. The solutions were evaporated nearly to dryness. The residue, if any, was taken up with the smallest possible quantity of water and a few drops of hydrochloric acid. 30–40^{cc} of amyl alcohol were then added and the water was expelled by bringing the alcohol to the boiling (128°–130° C.) Eight or ten drops of benzyl chloride were added and the solution was evaporated to about 10^{cc}. A perforated crucible, an asbestus felt and pressure were used in filtering. The filtrate was transferred to a weighed platinum dish and the alcohol driven off by evaporation on a water bath. As soon as the solution became viscous water was added and the evaporation repeated. The residue was finally taken up with water and sulphuric acid was added in slight excess. This solution was evaporated to dryness and the residue ignited and weighed as sulphate. The amyl alcohol precipitate was dissolved and transferred to a weighed platinum dish, evaporated to dryness and heated in an air bath at a temperature of about 300° C. and weighed.

The solubility corrections, as determined by Gooch,† were introduced, viz: For every 10^{cc} of amyl alcohol solution, exclusive of washings, 0·00051 grm. was added to the weight of the insoluble chlorides in case the residue was potassium chloride, 0·00041 grm. in case it was sodium chloride and 0·00092 grm. in case both were present. Equivalent amounts were taken from the weight of the magnesium sulphate.

The results of these separations, including determinations in many cases of the amounts of magnesia carried by the chloride precipitates, are here given.

*Several determinations as phosphate were made which, though agreeing together, gave somewhat lower results than were obtained by weighing the magnesium as sulphate. This may be due to impurities in the chloride or, as is more probable, it may be owing to the slight solubility of the phosphate

† Am. Chem. Jour., vol. ix, p. 48.

	Weight of KCl taken. grm.	Weight of KCl found. grm.	Corr. weight of KCl found. grm.	Error in corr. weight of KCl found. grm.	Weight of MgO found in KCl. grm.
(1)	0·1076	0·1075	0·1079	0·0003 +	0·0002
(2)	0·1084	0·1080	0·1083	0·0001 —	trace
(3)	0·1083	0·1079	0·1083	0·0000	0·0001
(4)	0·1077	0·1071	0·1076	0·0001 —	0·0001
(5)	0·1082	0·1075	0·1080	0·0002 —	0·0002
(6)	0·1085	0·1079	0·1083	0·0002 —	not det.
(7)	0·1628	0·1613	0·1622	0·0006 —	0·0004
(8)	0·1630	0·1614	0·1622	0·0008 —	0·0003
(9)	0·0109	0·0106	0·0110	0·0001 +	not det.
(10)	0·0116	0·0114	0·0118	0·0002 +	0·0001

	Weight of MgO taken. grm.	Weight of $MgSO_4$ found. grm.	Corr. weight of $MgSO_4$ found. grm.	Corr. weight of MgO_4 found. grm.	Error in corr. weight of MgO found. grm.
(1)	0·0092	0·0278	0·0274	0·0091	0·0001 —
(2)	0·0094	0·0275	0·0272	0·0091	0·0003 —
(3)	0·0417	0·1249	0·1245	0·0415	0·0002 —
(4)	0·0421	0·1264	0·1259	0·0420	0·0001 —
(5)	0·0828	0·2482	0·2476	0·0825	0·0003 —
(6)	0·0835	0·2509	0·2503	0·0834	0·0001 —
(7)	0·1662	0·5008	0·4998	0·1666	0·0004 +
(8)	0·1661	0·4986	0·4977	0·1659	0·0002 —
(9)	0·0830	0·2492	0·2488	0·0829	0·0001 —
(10)	0·0834	0·2494	0·2490	0·0830	0·0004 —

	Weight of NaCl taken. grm.	Weight of NaCl found. grm.	Corr. weight of NaCl found. grm.	Error in corr. weight of NaCl found. grm.	Weight of MgO found in NaCl. grm.
(11)	0·1113	0·1114	0·1116	0·0003 +	not det.
(12)	0·1066	0·1061	0·1065	0·0001 —	0·0002
(13)	0·1066	0·1058	0·1063	0·0003 —	trace
(14)	0·1064	0·1056	0·1062	0·0002 —	0·0001

	Weight of MgO taken. grm.	Weight of $MgSO_4$ found. grm.	Corr. weight of $MgSO_4$ found. grm.	Corr. weight of MgO found. grm.	Error in corr. weight of MgO found. grm.
(11)	0·0836	0·2496	0·2493	0·0831	0·0005 —
(12)	0·0835	0·2504	0·2499	0·0833	0·0002 —
(13)	0·1663	0·4979	0·4973	0·1658	0·0005 -
(14)	0·1665	0·4977	0·4970	0·1657	0·0008 —

	Weight of KCl taken. grm.	Weight of NaCl taken. grm.	Weight of chlorides found. grm.	Corr. weight of chlorides found. grm.	Error in corr. weight of chlorides found. grm.	Weight of MgO found in chlorides. grm.
(15)	0·0544	0·0529	0·1066	0·1076	0·0003 +	trace
(16)	0·0545	0·0531	0·1068	0·1077	0·0001 +	0·0004
(17)	0·1087	0·1063	0·2131	0·2142	0·0008—	0·0002
(18)	0·1085	0·1063	0·2122	0·2134	0·0014—	trace
(19)	0·1083	0·1064	0·2118	0·2137	0·0010	not det.

	Weight of MgO taken. grm.	Weight of $MgSO_4$ found. grm.	Corr. weight of $MgSO_4$ found. grm.	Corr. weight of MgO found. grm.	Error in corr. weight of MgO found. grm.
(15)	0·0827	0·2487	0·2475	0·0825	0·0002—
(16)	0·0850	0·2495	0·2484	0·0828	0·0002—
(17)	0·1664	0·5000	0·4987	0·1662	0·0002—
(18)	0·1663	0·5007	0·4995	0·1665	0·0002 +
(19)	0·1660	0·5010	0·4987	0·1662	0·0002 +

These results speak for themselves. It is evident that we have here a fairly good quantitative method. The errors are generally negative. This is to be expected. The large negative errors in the weights of the insoluble chlorides, in experiments (26)–(28), are probably due to loss from manipulation. 0·2 grams of either the insoluble chlorides or magnesia are perhaps too large quantities to deal with.

10^{cc} of amyl alcohol hold 1·14 grm. of magnesium chloride in solution at 14° C. and 1·23 grm. at 50° C. In the majority of the experiments the residual volumes of alcohol were less than 10^{cc}. They might with safety have been further reduced, thus reducing the solubility corrections.

In experiments (7), (8), (13), (14), (18) and (19) two precipitations of the insoluble chlorides were made, decanting the liquid, washing the residue, redissolving it and repeating the precipitation. The advisability of this is questionable. A more complete separation may be thus effected but the possibilities of loss from manipulation seem to counterbalance this advantage. Almost the same degree of completeness of separation may be attained by redissolving the precipitate with as little water and acid as possible and repeating the precipitation in the original solution.

In dehydrating the mixed chlorides it was noticed that the nature of the precipitate depended much on the relative amounts of the magnesium and the insoluble chlorides. Roughly speaking, when the weight of magnesia is equal to or exceeds that of the insoluble chlorides, the precipitate is thrown out as a fine powder. When however the insoluble

chlorides are greatly in excess, the tendency to obey the laws in accordance with which they crystallize asserts itself and we have a more or less coarsely granular deposit. This is especially true in the case of sodium chloride. These facts have a bearing on the question of completeness of separation.

Consider the amounts of magnesia found in the alkalies. They bear no relation to the amount of magnesium chlorides involved. It seems probable that the magnesia there found has been carried mechanically, and that it is not there as the result of decomposition. How can we better explain the finding of magnesia in the insoluble chlorides of experiment (1) where but 9 mgrm. of magnesia were involved. Here it is essentially the result of inclusion.

The desirability of adding water to the amyl alcohol solution of magnesium chloride before completing the evaporation is to be emphasized. By so doing it is possible to expel the alcohol so completely that even those experiments where there was the maximum amount of magnesia gave no trouble from carbonization.

In adding sulphuric acid a great excess is to be avoided. In the above experiments about twice the amount demanded by theory was used. The result, in the end, is economy of time and less danger of loss. It is better to add sulphuric acid a second time and repeat the evaporation, ignition and weighing. In the experiments involving an amount of magnesium equivalent to 0·16 grm. of the oxide, the ignited sulphate was found in a few instances to increase its weight after a second treatment with sulphuric acid. The lesser amounts were almost without exception wholly converted into the sulphate by a single treatment.

In the course of the work it was found that amyl alcohol and concentrated hydrochloric acid mix in all proportions. Furthermore if amyl alcohol containing but a small quantity of the acid be boiled down to but a small fraction of its original volume the residue contains chlorine in some form. 30^{cc} of the alcohol, to which 0·2^{cc} of concentrated hydrochloric had been added, having been reduced to 2^{cc} gave a decided test for chlorine. This suggested the possibility that the use of benzyl chloride was unnecessary. For, if amyl alcohol mixes with the concentrated acid and on being evaporated retains more or less of it to the last, the conditions which would prevent the decomposition of magnesium chloride are maintained. The easy solubility of the anhydrous magnesium chloride is beyond question. Possibly then, in the above experiments, the addition of benzyl chloride was uncalled for as in every case hydrochloric acid had been previously added to the salt solutions. To decide the question a few separations were made

without the benzyl chloride. Otherwise the conditions and methods were the same.

	Weight of KCl taken. grm.	Weight of KCl found. grm.	Corr. weight of KCl found. grm.	Error in corr. weight of KCl found. grm.	Weight of MgO found in KCl. grm.
(20)	0·1079	0·1078	0·1082	0·0003 +	trace
(21)	0·1081	0·1074	0·1079	0·0002 —	trace
(22)	0·1081	0·1076	0·1080	0·0001 —	0·0004
(23)	0·1080	0·1074	0·1079	0·0001 —	0·0003
(24)	0·1080	0·1071	0·1079	0·0001 —	0·0004
(25)	0·1076	0·1061	0·1070	0·0006 —	0·0002

	Weight of NaCl taken. grm.	Weight of NaCl found. grm.	Corr. weight of NaCl found. grm.	Error in corr. weight of NaCl found. grm.	Weight of MgO found in NaCl. grm.
(26)	0·1582	0·1576	0·1583	0·0001 +	trace
(27)	0·1591	0·1576	0·1582	0·0009 —	trace

	Weight of MgO taken. grm.	Weight of $MgSO_4$ found. grm.	Corr. weight of $MgSO_4$ found. grm.	Corr. weight of MgO found. grm.	Error in corr. weight of MgO found. grm.
(20)	0·0419	0·1250	0·1245	0·0415	0·0004 —
(21)	0·0418	0·1262	0·1257	0·0419	0·0001 +
(22)	0·0835	0·2508	0·2504	0·0835	0·0000
(23)	0·0827	0·2498	0·2493	0·0831	0·0004 +
(24)	0·1665	0·4996	0·4987	0·1662	0·0003 —
(25)	0·1667	0·5005	0·4995	0·1665	0·0002 —
(26)	0·1669	0·5009	0·5001	0·1667	0·0002 —
(27)	0·1662	0·4980	0·4973	0·1658	0·0004 —

It is evident that the benzyl chloride is not needed, further that its presence in the solution is harmless.

Summary.—In separating magnesium chloride from the chlorides of sodium and potassium, the treatment is as follows: evaporate the solution nearly or quite to dryness. Dissolve the residue in as little water as possible and add a few drops of hydrochloric acid. Then add 30–40^{cc} of amyl alcohol and expel the water by bringing the alcohol to the boiling. Continue the boiling until the volume of the solution is reduced to 10^{cc} or even considerably less. In filtering it is of great advantage to use a perforated crucible and an asbestus felt and to filter under pressure. In case the total chlorides exceed 0·2 grm. it may be advisable to decant the liquid, wash the residue, redissolve and repeat the precipitation. If this be not done the precipitate should be redissolved with the least possible quantity of water, a few drops of hydrochloric acid added

and the precipitation repeated in the original solution. The filtrate is transferred to a weighed platinum dish and evaporated. Water is added before the alcohol has been expelled and the evaporation continued. The residue is dissolved in water. Sulphuric acid is added in slight excess. This solution is evaporated to dryness, the residue ignited and weighed and the treatment with sulphuric acid is repeated. The residue of insoluble chlorides may be transferred to the weighed perforated crucible and dried at a temperature below their melting points or it may be dissolved and the solution transferred to a weighed platinum dish, evaporated and the residue dried, as above, and weighed.

Chemical Laboratory, Trinity College,
Hartford, April, 1892.

Art. XV.—*The Great Shear-zone near Avalanche Lake in the Adirondacks;* by J. F. Kemp.

In July, 1889, the writer was in the region of Lake Champlain, accompanied by V. F. Marsters, while gathering notes upon the igneous dikes so plentiful along its shores.* Our attention was directed to the great trap dike which is recorded near Avalanche Lake in the Adirondacks, and we made a trip into the woods to examine it. Although, when viewed from a distance this rock mass appeared as an undoubted dike, yet as soon as it was examined in a hand specimen on the spot, it was pronounced at once to be no true intrusion, but to constitute a shear-zone,† or zone of wall rock, dynamically metamorphosed along a fault. Subsequent microscopic examination has corroborated this view and the case in point is an interesting and somewhat unique exhibition of this phenomenon.

Avalanche Lake, a small body of water, is the very head of one of the feeders to the Hudson River. The pass by which it is reached from the north is on the watershed which diverts the northerly drainage into the Ausable River. The pass opens directly into the Lake, which is fed by springs. It is a narrow cañon between vertical walls of massive rock similar to the better known and larger Indian Pass farther west. It is five miles into the woods from the Adirondack Lodge. On the

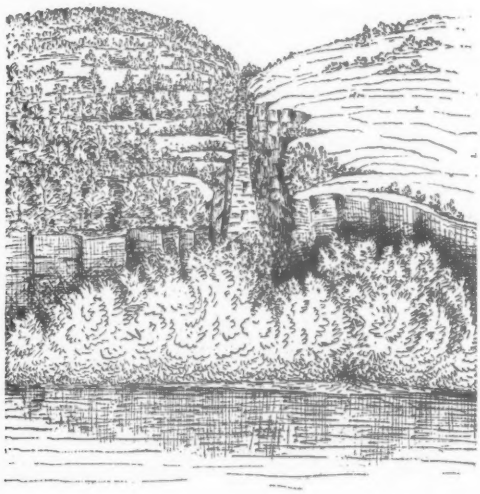
* A short digest of our results has appeared in the Transactions of the New York Academy of Sciences, vol. xi, p. 13, 1891. A full account will be printed elsewhere.

† For the use of this term compare Ch. Callaway, On the secondary minerals at shear zones, in the crystalline rocks of the Malvern Hills, Quart. J. Geol. Soc., Aug. 1889.

west of the lake is Mt. McIntyre, on the east are Mt. Colden and Avalanche Mts., which two are separated by the shear-zone. The twin mountain is also called Mt. McMartin. The lake and supposed dike were early discovered and recorded in connection with the mining enterprise that was started at Lake Henderson a few miles south.* The neighboring mountains, Colden, McMartin, etc., were named from the promoters of the mining enterprise. Redfield records that both the expeditions were accompanied by one of the recently appointed State geologists, the first by James Hall, and the second by E. Emmons.

The accompanying sketch has been traced directly from a photograph, taken from a point of view across the lake. It

1.



and the first micro-drawing were kindly made by Mr. Arthur Hollick. A sketch which but remotely resembles the original is given by Emmons in his report, p. 215 (also Ann. Rep. 1838, Atlas, plate 4.) The two peaks are dome-shaped knobs of massive rock, practically bare of vegetation and cleft from base to summit by the so-called dike. The latter forms a recess which is deeper at the lake than at the summit. At the

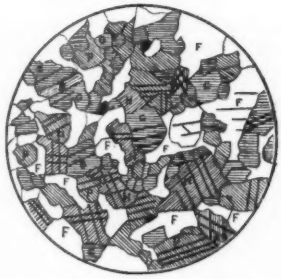
* W. C. Redfield: Some account of two visits to the Mountains of Essex Co., N. Y., 1836–37, etc., this Journ., I, xxxiii, 301. Second Ann. Rep. N. Y. State Survey, 1838, p. 225; Atlas plate IV. Emmons's Final Rep. on the 2nd Dist., p. 215.

lake it is 75 feet across. The walls are vertical. The so-called dike terminates in the lake where it is doubtless cut off by the cross fault that outlined the pass. It strikes N. 50–60 W.

The general rock of the mountains is a lighter colored variety of the plagioclase rock which is commonly but not always correctly called norite. Sections from the wall of the pass show coarse plagioclase in much the largest part, with which is probably a little orthoclase. The feldspar contains many minute inclusions which are probably small crystals of the dark silicates and iron ores, exhibiting thus a common character of the feldspars of rocks belonging to the gabbro family. The dark silicate is a brownish green hornblende of strong pleochroism. It occurs in shreds and irregular masses. Rather more abundant is a colorless enstatite of massive habit. The slide gives evidence of dynamic fractures and strains, but is far less broken than the rock of the shear-zone. The somewhat parallel arrangement of the hornblende occasions a very coarse gneissic structure. Under the microscope the enstatite follows a similar foliation. A slide from the north wall of the shear-zone exhibits much the same. It is chiefly plagioclase, with a little hornblende, enstatite and a little magnetite. The plagioclase has suffered some dynamic strains and shows faulting of twin lamellæ. Figure 2 is a drawing with the camera lucida. The heavy outlines mark the crystal boundaries as seen without crossed nicols, after which the twinning has been put in. The cleavages indicate the bisilicates, the rhombic is hornblende, the rectangular enstatite.

2.

3.



The rock of the shear-zone is dark gray in color and contrasted with the walls which are lighter. It thus resembles trap. In the hand specimen it is seen at once to be largely made up of garnets and this fact cast the first doubt on its igneous character. It also has the appearance of a rather mas-

sive hornblende schist or of a gneiss. Chips were taken from the edge of the zone and at intervals of ten feet across. They show some slight variations in mineralogical composition but they are essentially all the same rock. There is no evidence of the common chilling along the walls and coarser crystallization in the center, such as we see in dikes, but the structure is quite uniform throughout. The rock consists of broken irregular masses of plagioclase, 0·1–0·5^{mm}, of shreds of hypersthene, green, non-pleochroic, monoclinic pyroxene, pink garnet, greenish-brown hornblende, biotite and magnetite. The individual minerals are nearly in this order of abundance, but in the aggregate the dark silicates far surpass the feldspar. This is shown in fig. 3. The first four named, including plagioclase, are in excess. The plagioclase frequently shows abundant evidence of crushing and indeed as fig. 3, indicates all the minerals are irregular and broken. Fig. 3, was drawn with a camera lucida. The actual field is 4^{mm}. The impossibility of indicating in faithful line work the differences of the minerals has prompted putting initials on them. G is garnet; F, feldspar; A, amphibole; H, hypersthene; B, biotite and the hachures are uniform for each.

The hypersthene with pink to green pleochroism repeatedly passes into garnet which has a tint much like its pink. The same cracks run through both and it is not always possible to be sure when the pink shows, which mineral is present, until either the pleochroism or the isotropic character is proved. Two such are shown in fig. 3, one in the center, one in the lower right-hand quadrant. Pleochroic hypersthene passes in a similar way into non-pleochroic pyroxene. The same cleavage cracks pierce both, but the extinction angles are different, rising in the latter to various angles from 27°–44°. In instances the hornblende seems also to have resulted from the hypersthene. Biotite favors the neighborhood of magnetite, but in fig. 3 is shown cutting a garnet mass in two. It is comparatively rare.

It may be said, therefore, that in the walls we have a coarsely crystalline rock, containing plagioclase, hornblende and magnetite, but chiefly the first named. In the shear-zone, the dark silicates, hypersthene, green monoclinic pyroxene and garnet are collectively most abundant and with them is considerable plagioclase, a little hornblende, biotite and magnetite. The hypersthene passes into the monoclinic pyroxene and into garnet both of which are considered to be derived from it. That the feldspar has also helped to furnish these minerals, probably by contributions of calcium and aluminium, is a well-nigh irresistible conclusion from the way it is distributed in isolated patches through them. Dynamic effects are abundant.

The strip of the shear-zone may have been more basic originally than the walls, and may have contained more bisilicates or else infiltration has rendered it more basic. Alternations from light to dark so-called norite are not uncommon in the mountains, especially where the rocks are gneissoid. Being dynamically affected by faulting its minerals were crushed to the shreds in which we now see them and the secondary ones resulted. The hypersthene is regarded as an original mineral because it is abundant in the less disturbed districts and is a more distinctively igneous mineral than those derived from it. It cannot be denied, however, that the sheared zone may have been an original dike. If so its mineralogy and structure have been completely reorganized. So far as observations go, diabase dikes are quite plentiful in the outskirts of the mountains, but in neither of these characters does the shear-zone bear indication of ever having been diabase. The dynamic metamorphism of diabase has been shown in numerous instances in this country and abroad to yield amphibolites. The shear-zone is more like an eklogite than anything else. Moreover an intrusion of norite as a dike is a new phenomenon for this region whose noritic exposures are singular massive or gneissoid and send out so far as known, no dikes whatever. It is stated by Redfield that other and smaller dikes similar to the large one occur on Mt. McIntyre and frequently appear in the beds of brooks. He refers the lines of drainage to the greater relative decomposition of the supposed dikes. While no other one was seen by the writer which would be analogous to the shear-zone, these smaller occurrences ought to prove to be diabase or some similar rock if they really are intrusions.

A mineral aggregate similar to that of the shear-zone was met on Trembleau Point just south of Port Kent, and in the railways cuts. The country rock, which contains coarse plagioclase and monoclinic pyroxene as well as hypersthene, passes at times into dark, gneissoid bands, easily mistaken at first glance for trap which also seams the cuts in all directions. In the slides these bands are practically identical with the Avalanche shear-zone although at times containing more biotite. They make no recesses because freshly exposed.

A case of a shear-zone was also met at Hammondville in the No. 7 slope of the iron mines. The workings run down following a bed of magnetite, for 700–800 feet. They penetrate three narrow diabase dikes, by each of which the ore is faulted about 15 feet. The workings finally met another dark strip and the ore was cut off. A specimen was gathered under the impression that it was trap, but the section shows crushed remnants of the gneiss which is the wall rock. It is mostly chloritic alteration products, with shattered and strained grains

of quartz, sheared micas and recognizable fragments of plagioclase in the midst of decomposition material furnished by them. As the fault was in a different kind of rock from the shear-zones in norite, its products are somewhat different, and the great proportion of hydrated silicates give the impression that the shear-zones earlier described may be the result of a secondary metamorphism which the one at Hammondville has not experienced. This fault broke the ore and drill holes have failed to find it again. Experience in mining the adjacent Penfield bed has shown that the ore is cut off where the workings extend under a neighboring gully, and suggests that these topographical features are due in instances at least to faults. Such an explanation has proved true in many regions elsewhere* and will probably be applicable to some valleys of large size in the Adirondacks.

Crushed strips along faults are well known in mining districts and have often furnished the receptacles for ores, as at Butte, Montana.† The drift of modern opinion refers to them an increasing number of so-called fissure veins. Where conditions are unfavorable for vein formation dynamic metamorphism results. The work of Lossen, G. H. Williams, Teall and many others has shown the widespread effects of dynamic metamorphism, but a somewhat careful review has failed to reveal descriptions of any instances of such restricted effect and such mineralogical results as are exhibited on Avalanche Lake.

Geological Laboratory, Columbia College.

Art. XVI.—*On Herderite from Hebron, Maine;* by H. L. Wells and S. L. Penfield.

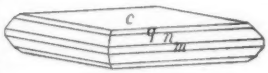
During the summer of 1891 we received from Mr. L. K. Stone of Paris, Maine, a small mineral specimen for identification. The unknown mineral consisted of a few yellowish white crystals on albite. They were about 3–5^{mm} in length and their total weight probably did not exceed 2 or 3 grams. Mr. Stone stated that this was the only specimen of the mineral that had been found, and that it was discovered in 1890 at the pollucite locality in Hebron.

A preliminary examination proved that the mineral was a phosphate, very slowly soluble in hydrochloric acid and yield-

*J. H. Kinahan, Valleys and their Relations to Fissures, Fractures and Faults. London: Trübner & Co.

†S. F. Emmons, Notes on the Geology of Butte, Trans. Inst. Min. Eng., July, 1887, and Structural Relations of Ore Deposits, idem. xvi, 804.

ing nearly neutral water in a closed tube. The latter reaction is entirely different from that of the Stoneham herderite, which gives off very acid water and etches the glass decidedly. Before the blowpipe in the platinum forceps it turned white, sprouted and fused at about 3 to a white enamel, coloring the flame very pale green. Only a few fragmentary crystals could be found which were suitable for measurement. The crystals had the habit shown in the accompanying figure. They were attached so that only the faces at one extremity of the macro-axis were developed and were grouped in nearly parallel position but with the basal planes slightly divergent.



The forms which were observed are:

c, 001, O; *m*, 110, I; *n*, 331, 3 and *q*, 332, $\frac{3}{2}$

The basal planes were irregular and yielded no satisfactory reflections of the signal, while *m* and *q* were small and frequently wanting. All of the faces except *c* were usually striated.

The measurements which were made are given below. The calculated values are derived from the axial ratio established by Prof. E. S. Dana* from the Stoneham herderite.

				Calculated.
$n \wedge n$,	$331 \wedge 33\bar{1} =$	45° 7′	*45° 10′	45° 26′	45° 6′
$n \wedge n$,	$331 \wedge \bar{3}31 =$	*102° 22′	103° 7′		103° 24′
$n \wedge m$,	$331 \wedge 110 =$	22° 14′	22° 53′		22° 33′
$n \wedge q$,	$331 \wedge 332 =$	16° 50′	16° 32′		17° 9′

Herderite was not suggested to us by the preliminary examination, and the measurements showed such a close relation to the angles of childrenite, that we fully expected the mineral to be some new variety of the latter, possibly a beryllium childrenite. It was not till the results of the quantitative analysis were obtained that we became aware of the true character of the mineral.

The similarity in crystalline form between childrenite and herderite is as follows:

Childrenite.		Corresponding forms on herderite.	
b,	010, i-$\bar{1}$	*c*,	001, O
	101, 1-$\bar{1}$	*m*,	110, I
p,	111, 1	*n*,	331, 3
s,	121, 2-$\bar{2}$	*q*,	332, $\frac{3}{2}$

Childrenite $a : b : c = \cdot7780 : 1 : \cdot5257$

Herderite in corresponding position $a : b : c = \cdot7752 : 1 : \cdot4929$

The axial ratio of the herderite was derived from the angles marked by asterisks in the above table of measurements. The

* This Journal, III, xxvii, 229.

angle $331 \wedge \bar{3}31$ could not be measured with certainty as the crystal, which was selected as best, gave double reflections from one of the faces. The angle 102° 22′ was selected as fundamental as it was from the brightest reflection, while 103° 7′ agrees better with the calculated angle of herderite.

The resemblance between the two minerals is remarkable, but since no chemical relation between them apparently exists, except that both are phosphates, the similarity in form may be accidental.

The whole specimen, except two or three small crystals, was sacrificed for a chemical examination. The material was so intimately mixed with albite that the greater part of it had to be separated by Thoulet's solution. The specific gravity of a pure crystal as shown by the heavy solution was 2·975, that of the Stoneham herderite was 3·006. The material used for analysis varied between 2·980 and 2·853.

A careful qualitative analysis showed the presence of beryllium oxide and the absence of all other bases except lime. The phosphoric acid determination was made in a separate portion by the molybdic acid method. The beryllium oxide and water were determined by methods analogous to that described by one of us for the analysis of herderite.* Unfortunately the determination of lime was a failure and there was not enough material for making a new determination. The fluorine was determined by weighing silicon fluoride, using a modification of Fresenius' apparatus.

The results of the analysis are as follows:

	Found.	Deducting Insol.	Calculated for $CaBeOHPO_4$.
P_2O_5	40·81	43·08	44·10
BeO	15·32	16·18	15·53
CaO†	[32·54]	[34·35]	34·78
H_2O	5·83	6·15	5·59
F	·40	·42	----
Insol.	5·27	----	----
	100·17	100·18	100·00

The analysis shows the mineral to be a new and interesting variety of herderite almost free from fluorine. The result confirms the idea advanced by Penfield and Harper that fluorine and hydroxyl are mutually replaceable in herderite, and the formula deduced by them for the mineral is confirmed.

Sheffield Scientific School, March, 1892.

* Penfield and Harper, this Journal, III, xxxii, 107.
† By difference.

Art. XVII.—*A method for the Iodometric Determination of Nitrates;* by F. A. Gooch and H. W. Gruener.

[Contributions from the Kent Chemical Laboratory of Yale College—XV.]

It has been shown by DeKoninck and Nihoul* that nitrates may be decomposed completely by the prolonged action of gaseous hydrochloric acid, and determined with accuracy by measuring the iodine set free when the products of decomposition, carefully kept from atmospheric contamination, act upon potassium iodide. These investigators recognized the difficulties attending the use of gaseous hydrochloric acid in analytical processes, and endeavored unsuccessfully to substitute the strong aqueous solution for the gaseous acid. The work to be described in the following account was performed in the search for a simpler method for the iodometric determination of nitrates.

According to a process recently developed in this laboratory† chloric acid may be determined with the greatest ease. It was shown that in the interaction of a chlorate with potassium iodide, arsenic acid, and sulphuric acid, in regulated quantities in aqueous solution and at the boiling temperature, the first action of the hydriodic acid set free from the iodide by the sulphuric acid is upon the chloric acid, and that not until this action is completed is the arsenic acid attacked and reduced with the simultaneous liberation of a corresponding amount of iodine. If the arsenic acid is taken in quantity sufficient to insure the final decomposition of the entire amount of iodide present, the arsenious acid found at the end of the action is an exact measure of the amount of iodide which escaped the action of the chlorate; and, the quantity of iodide originally taken being known, the amount acted upon by the chlorate, and so the amount of the chlorate itself, becomes known. The arsenious acid is determinable with great accuracy iodometrically, and the chief advantage of the process lies in the fact that the titration is made upon the residue, and that, no collection of the distillate being necessary, the sole apparatus employed in the process proper is an Erlenmeyer beaker and a bulbed tube hung in its neck as a trap to prevent mechanical loss.

This process we endeavored to apply to the determination of nitrates, but under none of the many variations of form and changes of conditions under which we tested it, were we able to secure complete decomposition of the nitrate without so

* Zeitschr. für angewandte Chemie, 1890, p. 477.
† Gooch and Smith, this Journal, vol. xlii, p. 220.

increasing the strength of the sulphuric acid that it was acted upon by hydriodic acid with the consequent unregistered escape of products of decomposition.

The endeavor to substitute a process essentially similar in principle, in which hydrochloric and antimonic acids should replace the sulphuric and arsenic acids proved likewise unsuccessful.

Abandoning therefore all attempts to so arrange the process that the oxidizing action of the nitrate should be registered in the residue, search was made for a reagent which should be capable of inducing easy decomposition of nitrates (after the manner of ferrous salts in acid solution) and yet (unlike ferrous salts) should be so readily restored to its primitive condition that the products of the oxidizing action of the nitrate should finally pass entirely to the distillate and be registered there. We have found the desired combination of qualities in manganous chloride dissolved to saturation in concentrated hydrochloric acid. This reagent is acted upon but slowly by nitrates at the ordinary temperature, but upon warming the nitrate begins at once to decompose with the formation of a higher chloride of manganese and liberation of nitric oxide. Ultimately if the heating is continued the chlorine of the higher chlorides is evolved and manganous chloride remains. During the process of heating the color of the solution passes from the original characteristic green through darker shades to black and returns by the reverse changes to the original tint. The decomposition of the nitrate extends under the conditions to the last traces, but the breaking up of the nitrates with the formation of the higher chloride, does not take place completely in the presence of water amounting to more than a third of the volume of the solution, and an action already established in strong acid is reversed by the addition of a large amount of water. Chlorates, peroxides, and other substances which liberate oxygen or chlorine when in contact with strong hydrochloric acid induce similar phenomena, but in the absence of such other substances the reaction serves to detect nitrates when present in fairly small amounts (perhaps one part in sixty thousand) as shown in the accompanying table:

KNO_3 taken.	$MnCl_2 . 4H_2O$ in strong HCl	Color developed.
0·01000 grm.	10 cm^3	Black.
0·00500	5	Black.
0·00100	5	Dark brown.
0·00050	5	Dark green.
0·00025	5	Deepened tint.
0·00015	5	Deepened tint.
0·00005	5	None.
0·00000	5	None.

In the first attempts to apply this reaction to the quantitative estimation of nitrates, 10 cm³ of the manganous mixture, the pure weighed nitrate, and an excesss of potassium iodide were put in a tubulated retort fitted with a hollow ground stopper drawn out at both ends so as to serve for the introduction of carbon dioxide evolved in a Kipp's generator charged with marble and acid previously boiled. The neck of the retort passed through a rubber stopper nearly to the bottom of a side-neck Erlenmeyer flask, used as a receiver, in the mouth of which the stopper fitted tightly. The side-neck of the receiver was joined by a rubber connector with a bent glass tube passing through a rubber stopper and reaching nearly to the bottom of a side-neck test tube into the mouth of which the stopper was fitted.

The first receiver contained hydrogen sodium carbonate in excess of the amount needed to neutralize the acid in the retort as it should distil over, a considerable quantity of potassium iodide (about 3 grm.) to aid in dissolving condensed iodine, and arsenious oxide in known amount and in excess of the quantity necessary to convert to hydriodic acid the free iodine evolved. The second smaller receiver was partly filled with a dilute solution of potassium iodide and hydrogen sodium carbonate. The current of carbon dioxide was started immediately upon introducing the contents of the retort, and the air was safely removed before the darkening of color, which begins to appear very soon, had spread through the liquid. Heat was applied, and the evolution of nitric oxide and later that of iodine began. The distillation was continued until nearly all the liquid had passed over. Finally, the contents of both receivers were united and titrated against decinormal iodine. The excess of arsenious oxide remaining unoxidized was taken as the measure of the iodine liberated and, accordingly, of the nitrate decomposed, upon the presumption that two molecules of the nitrate liberate ultimately six atoms of iodine according to the equation

$$2HNO_3 + 6H\text{-}I = 4H_2O + 2NO + 3I\text{-}I.$$

The choice of the solution for the retention of the halogen evolved was dictated by the consideration, in the first place, that very little iodine could pass through the alkaline arsenite to come into contact with the rubber stopper of the receiver on the way to the second absorbing liquid, and, secondly, that higher oxides of nitrogen reformed by the action of traces of air possibly introduced with the carbon dioxide or imperfectly removed by it could not liberate iodine from an iodide in in alkaline solution.

The iodide was introduced into the retort because having chosen to collect the halogen in alkaline solution it became necessary to take steps to break up, before it should reach the receiver, all nitrosyl chloride, the formation of which our experience in former lines of work not here detailed had led us to expect under the circumstances. In acid solution containing an iodide, nitrosyl chloride liberates iodine and is registered; in alkaline solution it breaks up with the formation of a chloride and a nitrite, the latter having no immediate action upon the arsenite. The results of experiments made in this manner are recorded in the accompanying table.

TABLE I.

KNO_3 taken.	KI in retort.	$MnCl_2$ mixture.	KNO_3 found.	Error.
0·1036 grm.	0·8 grm.	10 cm^3	0·1009 grm.	0·0027 grm. –
0·1083 "	0·8 "	10 "	0·1082 "	0·0001 " –
0·1064 "	0·8 "	10 "	0·1053 "	0·0011 " –
0·1068 "	0·8 "	10 "	0·1033 "	0·0035 " –
0·0551 "	0·8 "	10 "	0·0531 "	0·0020 " –

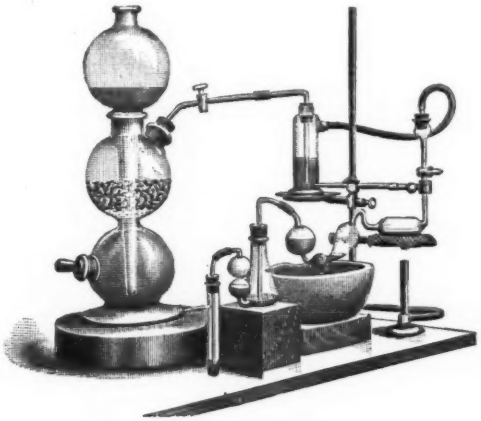
The experiments of Table II were carried out in a manner essentially similar to that of the experiments of Table I, excepting the single point that the iodine evolved in the process of decomposition of the nitrate was received in potassium iodide instead of in an alkaline arsenite. The contents of the receivers were united, made alkaline with hydrogen sodium carbonate, treated with an excess of decinormal arsenious acid, and the unoxidized arsenious acid was determined by decinormal iodine.

TABLE II.

KNO_3 taken.	KI in retort.	$MnCl_2$ mixture.	KNO_3 found.	Error.
0·2039 grm.	1·6 grm.	20 cm^3	0·2025 grm.	0·0014 grm. –
0·1060 "	0·8 "	10 "	0·1035 "	0·0025 " –
0·1036 "	0·8 "	10 "	0·1016 "	0·0020 " –
0·1013 "	0·8 "	10 "	0·1002 "	0·0011 " –
0·0521 "	0·5 "	10 "	0·0521 "	0·0000 " –
0·0235 "	0·5 "	5 "	0·0227 "	0·0008 " –
0·0273 "	0·5 "	5 "	0·0262 "	0·0011 " –
0·0136 "	0·2 "	5 "	0·0132 "	0·0004 " –
0·0011 "	0·2 "	5 "	0·0009 "	0·0002 " –

The errors of both sets of experiments, those of Table I and those of Table II, are considerable, all lie in the same direction, and are indicative of too low registering of the action of the nitrate, since of the complete decomposition of

the nitrate there can be no reasonable doubt in view of the proved behaviour of the manganese salt toward small amounts of nitrates. To us it seemed probable that the explanation of these results was to be sought in the failure of the titration in alkaline solution to indicate completely all the final products of the action of the nitrate. The formation, even in small amounts, of compounds of nitric oxide with iodine analogous to those which we know to be formed with bromine and chlorine, or the partial reoxidation of the nitric oxides by the action of iodine with the aid of water, an action which we recognize as possible under certain conditions of dilution, would account satisfactorily for the deficiency in the results of titration effected in alkaline solution. Upon this presumption the simple and obvious modification of titrating in acid solution should correct the error. Accordingly in the following series of experiments the plan of collecting the halogen and titrating in alkaline solution was abandoned, and since the addition of an iodide to the retort was no longer essential this practice was discontinued. The products of the action of the nitrate upon the manganese mixture—chlorine, nitric oxide and perhaps nitrosyl chloride,—were received directly in potassium iodide, and the iodine set free was titrated by sodium thiosulphate, itself standardized against iodine of known value with respect to a standard solution of decinormal arsenious oxide.



With the abandonment of the plan of putting the alkaline arsenite into the receiver the tendency of the iodine to pass onward to the second receiver is augmented and the possible action of the rubber stopper of the receiver becomes corre-

spondingly dangerous. We modified the apparatus, therefore, so that only glass should occur where by any possibility rubber connections might act upon the free halogen. In place of the ordinary retort we adopted a form of apparatus made use of formerly by one of us in the quantitative distillation of boric acid under the action of methyl alcohol—a pipette bent and fitted as shown in the figure. To this apparatus was sealed a Varrentrapp and Will nitrogen bulb, the exit tube of which was drawn out so that it might be pushed well within the inlet tube of the second receiver—a Will and Varrentrapp absorption flask—and held in place by an outside rubber connector. The third receiver acts simply as a trap to exclude air from the absorption apparatus proper. In conducting the experiment the receivers were charged with solutions of potassium iodide, the first containing three grams, the second one gram, and the third only a fraction of a gram for every tenth of a gram of nitrate used. The first receiver was kept cool during the process by immersion in water. The introduction of the nitrate and manganous mixture following it was made easy and safe by applying gentle suction to the end of the absorption train. The current of carbon dioxide was started immediately after putting in the manganous mixture, and after a suitable time had elapsed for the removal of air heat was applied to the retort and the distillation was continued until nearly all the liquid had passed over. Finally the contents of the receivers were united, the washing of the bulbs was effected easily and expeditiously by passing the wash-water directly through retort and receiver (the introduction of the manganese chloride into the distillate being not at all prejudicial to the accuracy of the titration), and the estimation of free iodine made by sodium thiosulphate as described. The results of the experiments conducted in this manner are given in Table III.

These results are fairly satisfactory. The mean error of the entire series is practically nothing. The manipulation is easy and rapid.

In brief, the process which gives us these results consists in the distillation of the mixture of the nitrate with a saturated solution of crystallized manganous chloride in strong hydrochloric acid in an atmosphere of carbon dioxide, the passage of the products of action into potassium iodide, and the titration of the liberated iodine by sodium thiosulphate. It is important to take precautions to prevent the contact of the free halogen with rubber stoppers or connectors, and any apparatus, suitable for ordinary quantitative distillation and absorption, which meets this condition will probably answer the requirements of the process. Our own preference is for the apparatus described and figured.

TABLE III.

KNO_3 taken.	$MnCl_4$ mixture.	KNO_3 found.	Error in terms of KNO_3.	Error in terms of HNO_3.
0·2038 grm.	20 cm^3	0·2047 grm.	0·0009 grm. +	0·0005 grm. +
0·2053 "	20 "	0·2057 "	0·0004 " +	0·0003 " +
0·1032 "	10 "	0·1035 "	0·0003 " +	0·0002 " +
0·1017 "	10 "	0·1004 "	0·0013 " —	0·0008 " —
0·1049 "	10 "	0·1049 "	0·0000 "	0·0000 "
0·1027 "	10 "	0·1023 "	0·0004 " —	0·0003 " —
0·0524 "	10 "	0·0526 "	0·0002 " +	0·0001 " +
0·0513 "	10 "	0·0512 "	0·0001 " —	0·0001 " —
0·0354 "	10 "	0·0350 "	0·0004 " —	0·0003 " —
0·0232 "	10 "	0·0230 "	0·0002 " —	0·0001 " —
0·0107 "	5 "	0·0106 "	0·0004 " —	0·0001 " —
0·0127 "	5 "	0·0130 "	0·0003 " +	0·0002 " +
0·0145 "	5 "	0·0143 "	0·0002 " —	0·0001 " —
0·0053 "	5 "	0·0052 "	0·0001 " —	0·0001 " —
0·0043 "	5 "	0·0047 "	0·0004 " +	0·0003 " +
0·0014 "	5 "	0·0018 "	0·0004 " +	0·0003 " +
0·0000 "	5 "	0·0000 "	0·0000 "	0·0000 "

The titration should be completed as soon as may be after admitting air to the distillate in order that traces of dissolved nitric oxide may not be reoxidized and again react upon the iodide present to liberate more iodine.

ART. XVIII.—*On some Alkaline Iodates;* by H. L. WHEELER. *With Crystallographic Notes;* by S. L. PENFIELD.

WHILE work on the compounds of iodine trichloride with alkaline chlorides* was in progress in this laboratory, it was noticed in making $KCl \cdot Cl_3I$, $RbCl \cdot Cl_3I$ and $CsCl \cdot Cl_3I$ that white crystals were often formed under certain conditions. These compounds proved to be $KCl \cdot KIO_3 \cdot HIO_3$, $RbCl \cdot HIO_3$ and $2CsIO_3 \cdot I_2O_5$. Since they were not analogous, although formed under similar conditions, and since the rubidium and cæsium salts have not been described, an investigation of them was undertaken. Attempts to prepare these compounds by other methods led to the discovery of several other iodates. The new compounds that have been prepared are as follows:

$RbIO_3$
$RbIO_3 \cdot HIO_3$
$RbIO_3 \cdot 2HIO_3$
$RbCl \cdot HIO_3$
$3RbCl \cdot 2HIO_3$
$CsIO_3$
$2CsIO_3 \cdot I_2O_5$
$2CsIO_3 \cdot I_2O_5 \cdot 2HIO_3$
$CsCl \cdot HIO_3$

* This Journal, xliv, 42.

The compound which separated from the solution of the potassium pentahalide has already been described, but since this is a new method of preparation and since there are conflicting statements concerning its state of hydration, it has been re-investigated.

The results of the investigation of the rubidium salts show that the normal iodate is the only one of the series that can be recrystallized unaltered from an aqueous solution. In the case of the cæsium compounds, the normal iodate and the salt $2CsIO_3 . I_2O_5$ are not decomposed by water. The other cæsium iodates give $2CsIO_3 . I_2O_5$ when recrystallized from water and not the normal iodate, thus showing an interesting difference between the rubidium and cæsium compounds.

It is the tendency of the acid rubidium iodates to separate in a higher state of hydration than the corresponding cæsium compounds.

It is also an interesting fact that the formation of the compounds of normal chloride and iodic acid was not observed on mixing the constituents. In the case of rubidium, products were obtained which proved to be $RbIO_3$, $RbIO_3 . HIO_3$ or $RbIO_3 . 2HIO_3$ according to the concentration of the solutions and the excess of RbCl or HIO_3. On the other hand by adding hydrochloric acid to a solution of rubidium iodate, if the acid is dilute $RbIO_3$, $2HIO_3$ is formed, if concentrated the iodate is completely decomposed. Similar experiments undertaken with cæsium chloride and iodic acid, did not give the peculiar double compound $CsCl . HIO_3$ but resulted, in each case, in the formation of $2CsIO_3 . I_2O_5$.

Method of Analysis.

After the substances were prepared for analysis as described in detail beyond, the halogens were determined by first reducing the solution of iodate with sulphur dioxide then precipitating with silver nitrate in the presence of nitric acid. This precipitate was then heated in a stream of chlorine, thus combining the test for chlorine and its determination in one operation. In the filtrate from the silver precipitate the alkali metal was determined as sulphate after the removal of the excess of silver by means of hydrogen sulphide. Oxygen was determined in a separate portion by precipitation with silver sulphate, drying the precipitate at 100° and then determining the loss on ignition. Duplicate halogen determinations were then made in this residue. In the case of the compounds containing the group I_2O_5, where an error would be introduced if the oxygen was determined in this manner, the substance itself was ignited and the oxygen calculated from the loss.

The presence of water in these compounds was determined by directly weighing it in a calcium-chloride tube, the substance being ignited in a combustion tube containing a mixture of lead chromate and lead oxide.

Normal rubidium iodate, $RbIO_3$.—This compound was made by adding one molecule of iodine pentoxide in either strong or dilute aqueous solution, to a solution of one molecule of rubidium carbonate. If the solutions are strong, the iodate separates as a sandy precipitate, but if they are hot and dilute it separates on cooling in small grains or as a crystalline crust. At 23° 100 parts of water dissolve 2·1 parts of the salt. The compound, after filtering on the pump, washing with a little water and drying on paper, gave the following results on analysis:

	Found.	Calculated for $RbIO_3$.
Rubidium,	32·17	32·82
Iodine,	48·50	48·75
Oxygen,	20·59	18·43

The salt when heated decrepitates strongly, melts, gives off oxygen but no iodine, and the residue is rubidium iodide. Hydrochloric acid readily dissolves it in the cold to a faint yellow-colored solution which increases in color on standing. On warming, chlorine is evolved and the solution turns bright yellow from the formation of iodine trichloride. If boiled with strong hydrochloric acid, RbCl . ClI* is formed which separates on cooling.

The formation of normal rubidium iodate was also observed: when a hot dilute aqueous solution of iodine trichloride was treated with rubidium carbonate. The compound thus obtained gave 48·43 per cent of iodine on analysis. Also by dissolving the acid iodate in a strong hot solution of rubidium chloride and allowing the mixture to crystallize. This was identified by a rubidium determination which gave 32·58 per cent. In general the iodates of rubidium all give this body when they are dissolved in hot water and the solutions left to crystallize. The products obtained in this manner decrepitated on heating and did not give off iodine. A rubidium determination in the substance obtained from RbCl . HIO_3 gave 32·76 per cent; from 3RbCl . 2HIO_3, 32·22 per cent.

Acid rubidium iodate, $RbIO_3HIO_3$.—This was obtained by mixing warm solutions of one molecule of iodine pentoxide and two molecules of rubidium chloride. The compound generally separates on cooling as a heavy crystalline powder. It is difficultly soluble in cold water. Hot water dissolves it more readily and on cooling, the normal iodate separates. It

* This Journal, xliii, 475.

is insoluble in alcohol. The crystals were filtered on a pump and washed with a little cold water and then pressed on paper. An analysis of these dried at 100° gave the following results, the oxygen being determined by difference.

		Calculated for $RbIO_3 . HIO_3$.
Rubidium,	20·13	19·58
Iodine,	58·12	58·19
Oxygen,	21·46	21·99
Hydrogen,	·29	·23

The reaction which takes place in the preparation of this compound is probably according to the following equation :

$$RbCl + 2HIO_3 = RbIO_3 . HIO_3 + HCl,$$

The hydrochloric acid thus liberated reacts on a part of the iodic acid, chlorine is evolved and the solution becomes yellow. When heated it does not decrepitate, but melts to a yellow mass, gives off water, then iodine and finally froths with the evolution of oxygen. The residue consists of rubidium iodide.

Diacid rubidium iodate, $RbIO_3 . 2HIO_3$.—For the preparation of this compound 5 grams of $RbIO_3$ were dissolved in 50 cc. of water with the aid of heat, then 13 grams of iodine pentoxide in 50 cc. of water were added, the mixture boiled down to half its volume and allowed to cool. The body separates as a heavy crystalline powder. It is difficultly soluble in cold water. When dissolved in hot water and the solution left to crystalline $RbIO_3$ separates. The product obtained as stated above was separated from the mother liquor by filtering on the pump, washed with a little cold water and dried at 100°.

	Found.		Calculated for $RbIO_3 . 2HIO_3$.
Rubidium,	13·93	14·13	13·96
Iodine,	61·91	62·48	62·20
Oxygen,	23·74		23·51
Hydrogen,	·42		·33

This compound does not lose water at 100°. When heated it does not decrepitate, but melts, gives off water, then iodine and oxygen, leaving a residue of rubidium iodide. The compound was also obtained by adding 10 cc. of hydrochloric acid sp. gr. 1·1 to 5 grams of $RbIO_3$ in 20 cc. of water. The mixture was warmed until all the $RbIO_3$ dissolved. It gave a faint yellow solution which slowly deepened in color. On standing, a well crystallized product of the compound under consideration was obtained containing 14·13 per cent of rubidium and 62·19 per cent of iodine.

The addition of a saturated solution of rubidium chloride to syrupy iodic acid produces a precipitate which dissolves again in the excess of iodic acid. When more rubidium chloride is added, the whole being kept over a lamp, a point is reached where a precipitate begins to form in the hot solution. This is the compound in question. It was identified by a rubidium and an iodine determination. This gave 14·17 per cent of rubidium and 61·83 per cent of iodine.

$RbCl \cdot HIO_3$.—This salt can be made by simply allowing a saturated solution of $RbCl \cdot Cl_3I$ to stand for some hours, when large colorless prisms form, attached to the plates of $RbCl \cdot Cl_3I$. The solution, after removing the crystals, warming to dissolve the pentahalide and pressing chlorine in again, does not yield a further deposit of the substance. This is explained by the fact that so much hydrochloric acid is formed in the solution that the formation of this compound is prevented. The crystals remain unaltered on exposure to the air. On treatment with cold water they are decomposed, losing their luster and becoming white. The solution has an acid reaction towards litmus. The hot saturated solution of this compound gives the normal iodate on cooling. The material for analysis was mechanically separated from adhering $RbCl \cdot Cl_3I$ and dried in the air.

	Found.		Calculated for $RbCl \cdot HIO_3$.
Rubidium,	28·88		28·78
Iodine,	42·29	42·62	42·76
Chlorine,	12·09	12·13	11·95
Oxygen,	16·33		16·16
Hydrogen,		·26	·33

This salt can also be prepared by adding a strong aqueous solution of rubidium hydrate to a strong solution of iodine trichloride in water. This gives at first a precipitate of the compound $3RbCl \cdot 2HIO_3$, and the solution left at rest for a few days gives the large well developed crystals of $RbCl \cdot HIO_3$ unmixed with $RbCl \cdot Cl_3I$. These were identified by their crystalline form.

On warming the crystals with hydrochloric acid $RbCl \cdot Cl_3I$ is formed, probably according to the following equation:

$$RbCl \cdot HIO_3 + 5HCl = RbCl \cdot Cl_3I + 3H_2O + Cl_2$$

and the $RbCl \cdot Cl_3I$ on further heating gives $RbCl \cdot ClI$ with the liberation of chlorine. When the substance is heated it melts, gives off water, chloride of iodine, and oxygen the residue consists of rubidium chloride and iodide. A determination of the halogens in this residue gave 3·52 per cent of chlorine and 53·66 per cent of iodine.

3RbCl . 2HIO$_3$.—This compound, which is analogous to the sodium compound 3NaCl . 2NaIO$_3$. 9H$_2$O described by Rammelsberg* and also to the salt 3NaI . 2NaIO$_3$. 19H$_2$O obtained by Penny† or 3NaI . 2NaIO$_3$. 20H$_2$O according to Marignac,‡ except that it contains no water of crystallization, was prepared by two methods. It was obtained by the addition of a hot, strong aqueous solution of rubidium hydroxide to a strong solution of iodide trichloride, the latter being in excess. The mixture was then filtered hot and on cooling, a mass of fine needles separated. The mother liquor on standing yielded the large crystals of RbCl . HIO$_3$. The needles are stable in the air and at 100°. From the hot saturated aqueous solution of the compound the normal iodate separates on cooling.

The formation of this compound was also observed on adding a strong solution of rubidium carbonate to a hot saturated solution of RbCl . Cl$_3$I, the latter being in excess. The colorless slender transparent needles thus obtained generally separate in groups radiating from a point on the surface of the yellow crystals of RbCl . Cl$_3$I. After separating the colorless crystals mechanically from the pentahalide they were air-dried on paper and then analyzed, while the material obtained according to the previous method was dried at 100°.

	From RbOH and ICl_3.		From Rb_2CO_3 and RbCl . Cl_3I.		Calculated for 3RbCl . 2HIO$_3$.
Rubidium,	35·41	34·58	35·78		35·87
Iodine,	35·27	36·00	35·87	35·81	35·52
Chlorine,	14·99	14·82	15·26	15·16	14·90
Oxygen,		13·15		13·64	13·43
Hydrogen,		·29		·30	·28

When heated, the substance does not decrepitate but melts, gives off chloride of iodine and the residue consists of a mixture of rubidium chloride and iodide. A sample of this residue gave on analysis 9·68 per cent of chlorine and 38·91 per cent of iodine.

Normal Cæsium iodate CsIO$_3$.—This was prepared by adding a moderately strong aqueous solution of iodic acid to a strong solution of cæsium carbonate, care being taken to have the carbonate in excess. When all the iodic acid had been added, the solution was boiled. On cooling, a crystalline mass separated consisting apparently of small cubes. At 24°, 100 parts water dissolve 2·6 parts of the salt. It is insoluble in alcohol. The body was prepared for analysis by filtering on the pump, washing with cold water and then pressing on paper and drying at 100°.

* Pogg. Ann., xliv, 548; cxv, 584.
† Ann. Ch. Pharm., xxxvii, 202. ‡ Jahresb., 1857, 124: Ann. Min., V, ix, 1.

			Calculated for $CsIO_3$.
Cæsium,	43·08	43·53	43·18
Iodine,	40·84		41·23
Oxygen,	15·74		15·59

This was also obtained in attempts to prepare a cæsium salt corresponding to $3RbCl . 2HIO_3$ by adding cæsium hydrate or carbonate in moderately strong aqueous solution to a strong solution of iodine trichloride in excess, when it at once separated in the form of a white sandy precipitate, which under the microscope was seen to consist of transparent grains of indefinite form. Unless the iodine trichloride is nearly saturated with the carbonate, $CsCl . Cl_2I$ or $CsCl . ClI$* is obtained mixed with the iodate. An iodine and oxygen determination in the air-dried salt gave 40·55 and 40·83 per cent of iodine and 15·67 per cent of oxygen. When this iodate is heated it does not give off iodine but melts and evolves oxygen. The residue is cæsium iodide.

$2CsIO_3 . I_2O_5$.—This substance can be prepared in pure condition and in large quantity by mixing a moderately dilute aqueous solution of two molecules of cæsium chloride with one molecule of iodine pentoxide dissolved in a little water. Any precipitate that may have been produced is dissolved by the aid of heat and more water if necessary. On cooling, the compound separates as a sandy powder. This can be washed with water or recrystallized from hot water without decomposition. It can also be recrystallized from dilute solutions of iodic acid. At 21° 100 parts of water dissolve 2·5 parts of this salt. It is insoluble in alcohol. The material for analysis was air-dried after pressing on paper.

	Found.	Calculated for $2CsIO_3 . I_2O_5$.
Cæsium,	27·93	28·00
Iodine,	53·42	53·47
Oxygen,	18·69	18·53

This compound invariably separates along with the crystals of $CsCl . Cl_2I$, when the latter is prepared in the absence of hydrochloric acid. The yield is not very large. It is thus obtained in the form of small rounded white nodules which on close inspection are seen to occur in pairs, the two nodules being on opposite sides of a thin layer of the pentahalide. They were mechanically separated from the pentahalide, no water being used to wash the compound when prepared for analysis. The following results are sufficient for its identification.

* This Journal, III, xliii, 17, and xliv, 42.

Cæsium	29·11
Iodine	50·21
Oxygen	18·99
Chlorine	3·24

This compound was also obtained by the following methods. By mixing 6 grams of $CsIO_3$, 20 cc. of water and 10 cc. of HCl sp. gr. 1·1. The mixture was boiled, it became yellow and chlorine was evolved. When cooled the substance separated as a crystalline crust. It was identified by a determination of cæsium which gave 28·40 per cent.

The compounds $2CsIO_3 . I_2O_5 . 2HIO_3$ and $CsCl . HIO_3$ give this body when their hot saturated solutions are cooled. A cæsium determination in the products thus obtained gave 27·94 and 28·12 per cent respectively.

When this body is treated with hydrochloric acid, sp. gr. 1·1, the solution becomes yellow, evolves chlorine on warming, and when concentrated on the water bath yields on cooling well crystallized $CsCl . ClI$. Analysis gave 50·63 per cent of cæsium chloride, calculated for $CsCl . ClI$ 50·90 per cent.

When heated in a closed tube it gives no sign of water, gives off iodine then melts with the evolution of iodine and oxygen. The residue consists of cæsium iodide.

$2CsIO_3 . I_2O_5 . 2HIO_3$.—This body was obtained by adding 5 grams of $2CsIO_3 . I_2O_5$ to a boiling solution of 25 grams of iodine pentoxide in sufficient water to form a syrup. Water was then added and the precipitate thus produced proved to be the compound in question. Thus produced it separates as a finely divided amorphous precipitate which can be dried in the air or at 100° without losing water. It is difficultly soluble in water and when crystallized from an aqueous solution gives $2CsIO_3 . I_2O_5$. An analysis of the substance dried at 100° gave

	Found.	Calculated for $2C_3IO_3I_2O_5 . 2HIO_3$.
Cæsium,	19·71	20·43
Iodine,	57·68	58·52
Oxygen,	20·41	20·89
Hydrogen,	·12	·16

Water determinations in samples dried in the air on paper gave 1·45 and 1·38 per cent; theory requires 1·44.

When the substance is heated it gives off water and iodine, then oxygen, the residue consisting of cæsium iodide.

$CsCl . HIO_3$.—This was obtained in an attempt to increase the yield of $2CsIO_3 . I_2O_5$ by adding a rather small quantity of cæsium carbonate to a hot saturated solution of $CsCl . Cl_3I$, when on cooling and allowing the mixture to stand, colorless,

flat, transparent prisms separated on the yellow crystals of $CsCl \cdot Cl_3I$ previously formed. These colorless prisms were picked out from the solution dried on paper and separated mechanically, as far as possible from any adhering $CsCl \cdot Cl_3I$. These on analysis gave the following results.

	Found.		Calculated for $CsCl \cdot HIO_3$.
Cæsium	38·09		38·60
Iodine	36·08	36·29	36·86
Chlorine	11·69	11·82	10·31
Oxygen	13·85		13·94
Hydrogen	.30		·29

The crystals remain unaltered on exposure to dry air but on treating them with water they immediately become opaque. On recrystallizing from water they give $2CsIO_3 \cdot I_2O_5$. When the substance is heated it gives off water and iodine chloride, melts, and gives off oxygen, the residue consisting of chloride and iodide of cæsium. When it is warmed with hydrochloric acid it undergoes the same decomposition as the corresponding rubidium compound.

$KCl \cdot KIO_3 \cdot HIO_3$.—This compound has previously been prepared by treating KIO_3 with hydrochloric acid, or a solution of iodine trichloride with potassium hydrate or carbonate. It has been described by Serullas* and Rammelsberg† as anhydrous and the formula $2KCl \cdot 2KIO_3 \cdot I_2O_5$ was assigned to the salt. Millon‡ from his determination of potash in this salt concluded that the substance contained a molecule of water, but he made no determination of it. Finally, Marignac§ who examined it more carefully, made a determination of the water by drying the substance at 100°, then igniting it in a tube with metallic copper and collecting, and weighing the water by means of a sulphuric acid tube.

The compound obtained from a solution of $KCl \cdot Cl_3I$ separated in shining transparent prisms, stable in the air. It contained water corresponding to the formula $2KCl \cdot 2KIO_3 \cdot I_2O_5 \cdot H_2O$ or $KCl \cdot KIO_3 \cdot HIO_3$. An analysis of the air-dried salt gave the following results.

	Found.		Calculated for $KCl \cdot KIO_3 \cdot KIO_3$.
Potassium	16·94	16·83	16·82
Iodine	54·46		54·66
Chlorine	7·72		7·64
Oxygen			20·66
Hydrogen	·20		·22

* Ann. Ch. Phys., II, xliii, 113. † Ann. Ch. Phys , III, ix. 407.
‡ Pogg. Ann., xcvii. § Jahresb., 1856, 298. Ann. Min., V, ix, 1.

This compound and the one obtained by Marignac are therefore identical.

On ignition it gives off water, iodine chloride and oxygen, the residue consisting of potassium iodide and chloride. An analysis of this residue gave 2·39 per cent. chlorine and 70·87 per cent. iodine.

The author takes occasion here to express his obligations to Professor H. L. Wells for the use of the material in this investigation and for valuable suggestions, also to Professor S. L. Penfield who has kindly furnished the crystallographical descriptions.

Notes on the Crystalline form of RbCl . HIO_3 and CsCl . HIO_3; by S. L. Penfield.

RbCl . HIO_3.

1.

The form of RbCl . HIO_3 is monoclinic. The crystals are highly modified, doubly terminated prisms, fig. 1. The faces gave fair reflections and the measurements which were chosen as fundamental are marked by an asterisk in the table of angles.

The axial ratio and forms are as follows:

$$\dot{a} : \bar{b} : \dot{c} = 0{\cdot}9830 : 1 : 0\,7577, \quad \beta = 100 \wedge 001 = 87^\circ\ 56'$$

a, 100, *i*-$\bar{i}$	*l*, 320, *i*-$\bar{\frac{3}{2}}$	*d*, 011, 1-$\dot{i}$	*g*, $\bar{1}02$, $\frac{1}{2}$-$\bar{i}$	*q*, 142, -2-$\dot{4}$
b, 010, *i*-$\dot{i}$	*m*, 110, I	*e*, 101, -1-$\bar{i}$	*o*, 211, -2-$\bar{2}$	*s*, $\bar{2}11$, 2-$\bar{2}$
c, 001, 0	*n*, 120, *i*-$\dot{2}$	*f*, $\bar{1}01$, 1-$\bar{i}$	*p*, 111, -1	*u*, $\bar{1}11$, 1.

		Measured.		Calculated.				Measured.		Calculated.	
$a \wedge c$,	$100 \wedge 001$	=*87°	56′			$a \wedge d$,	$100 \wedge 011$	= 88°	29	88°	21′
$a \wedge e$,	$100 \wedge 101$	=*51	5			$a \wedge s$,	$\bar{1}00 \wedge \bar{2}11$	= 38	32	39	47
$c \wedge d$,	$001 \wedge 011$	=*37	8			$a \wedge u$,	$\bar{1}00 \wedge \bar{1}11$	= 59	57	59	38
$a \wedge l$,	$100 \wedge 320$	= 33	13	33°	13′	$e \wedge p$,	$101 \wedge 111$	= 30	28	30	31
$a \wedge m$,	$100 \wedge 110$	= 44	7	44	29½	$f \wedge u$,	$\bar{1}01 \wedge \bar{1}11$	= 31	22½	31	24
$a \wedge n$,	$100 \wedge 1\bar{2}0$	= 62	42	63	1½	$p \wedge q$,	$111 \wedge 142$	= 26	36	26	30
$a \wedge o$,	$100 \wedge 211$	= 38	19	38	28½	$c \wedge g$,	$001 \wedge \bar{1}02$	= 21	17	21	20
$a \wedge p$,	$100 \wedge 111$	= 57	13	57	14	$c \wedge f$,	$001 \wedge \bar{1}01$	= 38	26	38	23½

CsCl . HIO_3.

The form of CsCl . HIO_3 is monoclinic. The crystals, from the one crop which was examined, were about 5 mm. in length and had the habit shown in fig. 2. They were attached at one end and usually grew in radiating and divergent groups. The faces were not very perfect and only approximate measure-

2.

ments could be made. Those which were chosen as fundamental are:

$m \wedge m$, $110 \wedge \bar{1}10 = 90° 12'$ $m \wedge p$, $110 \wedge 221 = 24° 37'$ $a \wedge p$, $100 \wedge 221 = 49° 53'$

The axial ratio and forms are as follows:

$\dot{a} : \bar{b} : \dot{c} = 0{\cdot}9965 : 1 : 0{\cdot}7698$ $\beta = 100 \wedge 001 = 89° 53\frac{1}{2}'$

a, 100, i-$\bar{i}$	m, 110, I	d, 403, $-\frac{4}{3}$-$\bar{i}$	s, $\bar{4}03$, $\frac{4}{3}$-$\bar{i}$	p, 221, —2
c, 001, 0	n, 130, i-$\dot{3}$	e, 203, $-\frac{2}{3}$-$\bar{i}$	u, $\bar{2}03$, $\frac{2}{3}$-$\bar{i}$	o, 263, -2-$\dot{3}$

The pyramids p and o were frequently wanting. The orthodomes d, e, s and u were very constant in their development and gave to the crystals an orthorhombic habit. Owing to the curved and striated character of the faces the symmetry could not be satisfactorily determined by measurement, but the optical properties showed that the crystals were truly monoclinic. In polarized light the tables show an extinction parallel to the ortho- axis and in convergent light one of the optical axes and the acute bisectrix can be seen near the limits of the field. The plane of the optical axes is the clinopinacoid.

These two salts, although entirely different in crystalline habit, are very similar in their axial ratios.

Sheffield Scientific School,
April, 1892.

ART. XIX.—*Development of the Brachiopoda.* Part II. Classification of the Stages of Growth and Decline; by CHARLES E. BEECHER. (With Plate I.)

A BRIEF review of the known embryology of the Brachiopoda is desirable, in order to account for some of the differences presented by adult forms in the several divisions of the class. This knowledge is far from complete, and is confined to a few species, but much of interest bearing on the later development of the organism may be obtained.

The important memoirs of Morse,*[18, 19] Kovalevski,[15] Lacaze-Duthiers,[16] and Shipley,[22] contain nearly all that is known regarding the early embryology of brachiopods. The genera included in the works of these authors comprise Cistella, Terebratulina, Liothyrina, and Lacazella. Later larval stages of the genus Glottidia have been fully described by Brooks.[4] Müller,[20] also, has given a description and figures

*The works referred to by numbers are cited in full in the list appended to this article.

of a larval form doubtfully referred to Discinisca. The results of these observers must at present be taken without reservation, and are thus made use of in the present paper.

Something is known, therefore, of the early stages in each of the four groups or orders proposed by the writer.[1] The Atremata, Neotremata, and Protremata are represented by a single genus only in each; Glottidia, Discinisca, and Lacazella, respectively; and the Telotremata, by Cistella, Terebratulina, and Liothyrina. Were Glottidia and Discinisca as well known as Cistella, Terebratulina, and Lacazella, some comparisons could undoubtedly be made which would enlighten many obscure points of anatomy and morphology, as well as give clearer insight into the history and origin of each group.

Cistella and Terebratulina are taken as standards of the embryological development on account of the completeness with which they have been studied, and because their points of difference are not great. Lacazella shows such peculiar features, that its history must be discussed separately. The nepionic Glottidia and Discinisca, too, present characters which evidently had an early history somewhat different from Cistella or Terebratulina.

In taking up the review of the observed stages of growth, an attempt will be made to fix their limitations. To this end the admirable nomenclature proposed by Hyatt[9, 10] is here adopted, as it is more convenient and of wider application and significance than the terms heretofore used. Thus far this system has been employed principally in studies relating to the mollusca, and its application to the Brachiopoda will necessarily require some illustration and explanation. In the preface to "Genesis of the Arietidæ," Hyatt has presented a summary of the theoretical opinions resulting mainly from his studies in the Cephalopoda. It is believed that nearly the same ground may be covered in the Brachiopoda, and thus the truth of these deductions will receive further evidence from another class of organisms.

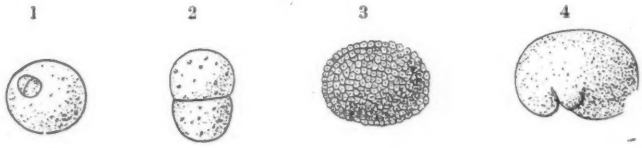
Embryonic stages.

The true embryonic stages are classified by Hyatt as *Protembryo*, *Mesembryo*, *Metembryo*, *Neoembryo*, and *Typembryo*. To these Jackson[11] has added the *Phylembryo*, taking it from the later stages of the Typembryo to represent the period when the animal can be referred definitely to the class to which it belongs.

The succeeding stages in the growth of the animal to maturity are termed by Hyatt *nepionic* (young), *nealogic* (adolescent), and *ephebolic* (mature), while old age characters

are called *geratologic*. The subject of geratology is further divided into the *clinologic* and *nostologic* stages.

The application of this nomenclature of the stages of growth and decline to the Brachiopoda is shown on the following pages.



Cistella neapolitana Scacchi.

FIGURE 1.—Protembryo. Unsegmented ovum.
FIGURE 2.—Protembryo. Ovum composed of two spheres.
FIGURE 3.—Mesembryo. Blastosphere.
FIGURE 4.—Metembryo. Gastrula. (1-4, after Shipley)

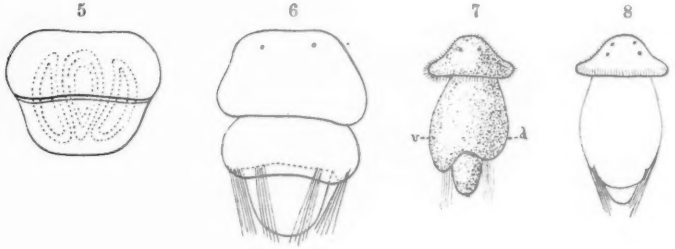
The *Protembryo*, as in other groups of organisms, includes the ovum and its segmented stages preceding the formation of a blastula cavity. Figures 1, 2, show protembryonic stages of Cistella. The eggs are spherical, pyriform or ovoid, and the segmentation proceeds in a regular manner, resulting in a blastosphere composed of equal parts.

The *Mesembryo*, or blastosphere, figure 3, has been observed in Cistella, Terebratulina, and Lacazella. The blastula cavity is small.

The *Metembryo*, or gastrula stage, figure 4, is developed from the blastosphere in two ways; (*a*) by embolic invagination in Cistella and Terebratulina (Kovalevski and Shipley), and (*b*) by delamination in Lacazella (Kovalevski). At the close of this stage, the archenteron in Cistella is trilobed, consisting of a central cavity, or mesenteron, connecting on each side with the body cavity.

The *Neoembryo*, represented by the trochosphere and segmented ciliated cephalula stages, has been more fully observed than any of the preceding. The first advance from the completed gastrula is in the separation of the mesenteron from the body cavity, and the division of the organism into two segments or lobes, the cephalic and caudal, figure 5. Later a third or thoracic segment is developed and carries four bundles of stiff barbed setæ, figure 6. The cephalic and caudal lobes are densely ciliated. During the subsequent cephalula period, two eyes, then two others appear in Cistella, and at the same time the dorsal and ventral sides of the thoracic segment become extended over the caudal, and are progressively defined as two lobes, figures 5–9, 24, 25.

Terebratulina has a tuft of bristles on the top of the cephalic segment. In Lacazella, the bundles of setæ are absent, and the head is more distinctly differentiated from the anterior segment than in Cistella. The closing cephalula stage in Cistella has an umbrella-like expansion of the cephalic border, and the organism becomes a free swimming larva, figures 7–9.



Cistella neapolitana Scacchi.

FIGURE 5.—Neoembryo. Embryo of two segments.
FIGURE 6.—Neoembryo. Cephalula, ventral side; showing cephalic, thoracic, and caudal segments, eye spots, and bundles of setæ. (5, 6, after Kovalevski.)
FIGURE 7.—Neoembryo. Lateral view of completed cephalula stage; showing extent of dorsal (*d*) and ventral (*v*) mantle lobes, and umbrella-like cephalic segment.
FIGURE 8.—Neoembryo. Same stage; ventral view. (7, 8, after Shipley.)

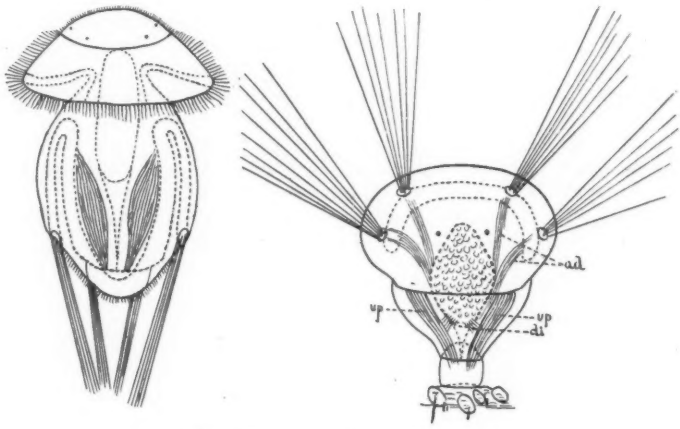
Larval stages.

The *Typembryo* is the larval stage at which some distinctive features make their appearance, but before the special characters of the class are to be found, figure 10. It is analogous to the molluscan embryo in which a shell gland and plate-like initial shell are developed. There is, however, no homology of parts or organs between the typembryonic mollusk and brachiopod.

In Cistella and Terebratulina the development of the typembryo has been observed, and consists of the folding upwards of the lobes which have been developed from the thoracic segment to form the mantle, so that they gradually enclose the anterior end, figures 24–27. The surfaces of the mantle which were exterior in the cephalula have now become inner and the bundles of setæ have revolved 180°, changing their direction from posterior to anterior. This leaves the lower part of the thoracic and the whole of the caudal segment exposed. The outer surface of the mantle is invested with a hard integument, which, upon completion, and before the growth of the true shell forms the protegulum. The

pedicle at this stage is also defined, being a modification of the caudal segment. It may serve to attach the larva to foreign objects, as in Cistella, figure 10, and Terebratulina, or it may remain undeveloped for a time as in Glottidia and Discinisca. A rudimentary digestive tract is present.

9 10



Cistella neapolitana Scacchi.

FIGURE 9.—Neoembryo. Completed cephalula stage.

FIGURE 10.—Typembryo. Transformed larva resulting from folding upwards of mantle lobes over cephalic segment: *ad.* muscles from bundles of setæ to sides of body cavity; *di.* muscles from dorsal to ventral sides of body; *vp*, muscles from ventral side of body to caudal segment or pedicle. (9, 10, after Kovalevski.)

The body muscles which have been developed thus far consist of four distinct pairs. Two pairs lie close to the sides of the body cavity, and extend to the points of insertion of the bundles of bristles, figure 10, *ad.* They become after transformation the four adductor muscles of the valves. The third pair extends from the ventral side of the body to the caudal segment, and is converted into the ventral pedicle muscles, figures 10, 15, 16, *vp.* The fourth pair is situated posterior to the digestive tract, and extends from the dorsal to the ventral wall of the body, figure 10, *di.* They form the divaricator muscles in the mature brachiopod, figure 16, *di*, and are divided into or duplicated by a pair of dorsal and a pair of ventral divaricators. There is also a pair of dorsal pedicle muscles in the larva of Liothyrina and Terebratulina.

The folding upwards of the mantle lobes forms the first

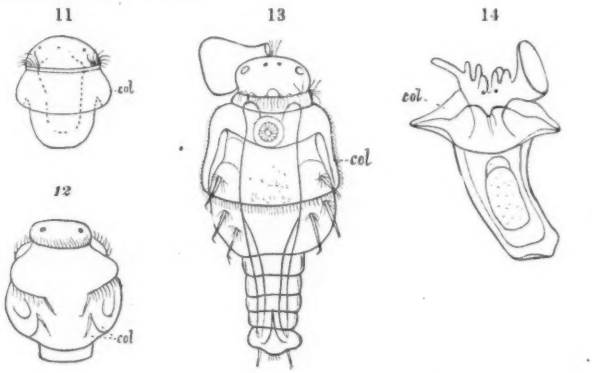
hinge line of the future valves, *hl*, figures 26, 27. Thus its origin is not, as in pelecypods, a line produced by the bending of a single plate (Jackson), but is the line along which the two mantle lobes are bent against the body. Between them projects posteriorly nearly half the body of the animal, and the whole opening corresponds to the pedicle opening of later stages of growth. The hinge of brachiopods, therefore, is not primarily a line of articulation of the valves, but the limiting borders between the body and the attached edges of the mantle. Secondarily, and during later growth, the extension of the valves along a line of apposition forms a true hinge line.

The first points of contact of the valves to form the true hinge lie adjacent to the right and left sides of the body of the animal, at the cardinal extremities, figure 15, *t*. Here naturally the first hinge teeth are formed, and their position corresponds to that in adult individuals; namely, on each side of the cardinal opening. The enlarging of the cardinal opening by shell growth results in the gradual divergence or separation of the teeth as in Terebratulina. In species with extended hinge lines, as in many forms of Spirifer, Orthis, and Strophomena, the teeth still lie in their original position on each side of the cardinal opening, and the elongation of the hinge has come not only from the enlargement of the opening by growth, but by additions at the hinge extremities, so that the teeth are situated on each side of the central area, below the beak, and not at the cardinal angles. The young of these genera, however, all have the hinge teeth at the extremities of the hinge, as the cardinal opening then occupies the whole posterior area of the shell.

Adult specimens of Kutorgina (*K. cingulata* Billings) have a deltidium as in Strophomena. The cardinal opening including the deltidium occupies the whole posterior end of the shell, and according to a statement made to the writer by Mr. Charles Schuchert, there are rudimentary teeth at the cardinal extremities. Therefore, this genus represents a nepionic condition of later forms, and, on account of these and other characters, it is believed to be related to Orthisina and Strophomena, of which it is the ancestral type. It consequently belongs to the articulate brachiopods.

The embryonic stages up to this point have frequently been compared to similar stages in other organisms, especially in the Annelida and Polyzoa. Without repeating these comparisons, which may be consulted elsewhere,[4, 12, 15, 16-19, 21] attention is called to the similarity of development of the brachiopod typembryo to the larval stages of Spirorbis. There are, how-

ever, important structural differences. An article by J. W. Fewkes, "On the Larval Forms of *Spirorbis borealis* Daudin,"[9] contains a nearly complete and very interesting account of the development of this chætopod. There is a striking resemblance in the characters of the cephalula stages in both organisms, as may be seen on comparison, figures 11, 12. Spirorbis develops a posteriorly directed extension from the middle segment, called a collar, which in later stages is reflexed anteriorly so as to cover more or less the cephalic portion, thus agreeing with the growth and change in position of the mantle in Cistella. The ventral lobe is also the larger in both. Many other comparisons and homologies have been made by Morse,[10] and the one here described is even more marked than his reference to the lobation of the cephalic collar in Sabella. Four figures are introduced illustrating the principal changes in Spirorbis. They may be compared with the development of Cistella shown in figures 6–10.



Spirorbis borealis Daudin.

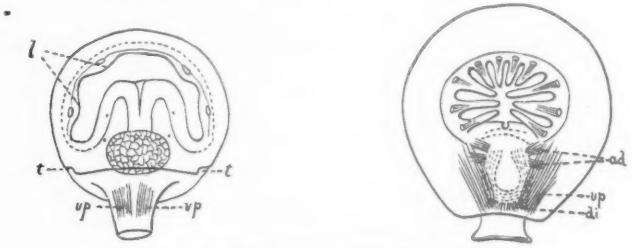
FIGURE 11.—Cephalula, developing lobe from the body (*col.*).
FIGURE 12.—More advanced stage.
FIGURE 13.—Larval form before transformation; showing posteriorly directed expansion (*col*) from thoracic segment.
FIGURE 14.—Transformed Spirorbis; showing folding upwards of collar partially enclosing head. (11–14, after Fewkes.)

It is not intended by this to indicate a close relationship with the chætopods, for the writer is inclined to accept the opinion of Joubin,[12] that the brachiopods constitute a distinct and independent class.

The *Phylembryo*, figure 15, differs from the Typembryo in (*a*) the completion of the embryonic shell, or protegulum; (*b*) the first appearance of the tentacular lobes of the lophophore,

or arms; (*c*) the usual dehiscence of the four bundles of setæ; (*d*) the obsolescence of the eyes; (*e*) the definition of the œsophagus and stomach; and (*f*) the agreement of the muscular system with that in adult forms. These features, with the pedicle which appeared in a preceding stage, represent the brachiopod phylum, and are properly referred to the phylembryonic period of Jackson. Although the molluscan stage called the prodissoconch in pelecypods, the protoconch in cephalopods and gastropods, and the periconch in scaphopods, represents the completed phylembryo of these groups, as the protegulum represents a like period in the developing brachiopod, yet there is no homology of distinctive organs.

15 16



Cistella neapolitana Scacchi.

FIGURE 15.—Phylembryo. Brachiopod; showing shell (protegulum), beginning of tentacles of lophophore (*l*), obsolescence of eye spots, and formation of œsophagus. *t*, hinge teeth; *vp*, ventral pedicle muscles.

FIGURE 16.—Nepionic brachiopod; showing distinct tentacles of lophophore, mouth and stomach, and transformation of muscles from typembryo, figure 10; *ad*, adductors; *di*, divaricators; *vp*, ventral pedicle muscles. (15, 16, after Kovalevski.)

The mantle of mollusks is first formed on the posterior dorsal side, and is in the shape of a disc, which gradually envelops the animal to a greater or less extent, and may become distinctly lobed. As has been shown, this organ in the brachiopods develops simultaneously from the dorsal and ventral side of the thoracic segment of the cephalula, and is primarily bilobed.

The initial shell of brachiopods is not produced from a distinct shell gland, as in the mollusca, but is an integument of the surface of the mantle lobes, and intimately connected with them. The position of the valves is dorsal and ventral. The pedicle has no organic similarity with either a foot or a byssus.

The mouth of mollusks (and annelids) is formed below the base of the cephalic lobe of the cephalula, and may be the blastopore, while in the brachiopods it is near the anterior pole

within the cephalic segment. Notwithstanding these differences, so many parts are functional equivalents, that their growth and development may be discussed and interpreted in the same terms.

Before passing to later stages of growth which become more and more divergent from a common simple type, some points previously omitted, relating to Thecidium (Lacazella), Lingula (Glottidia), and Discinisca, should be here noted. As Lacazella is a form in which the ventral valve in the nealogic and ephebolic stages is cemented to foreign objects by calcareous fixation, it bears about the same relation to other brachiopods that Ostrea bears to Avicula, among the pelecypods, and a corresponding early absence, or modification, of many features present in adult individuals should be looked for. From what is known of the geological history of Thecidium, and if the interpretations of its phylogeny by the writer are correct, it is derived from an ancestry which had a similar condition of fixation as early as the Upper Silurian. Thecidium is apparently not a terebratuloid genus. Its structural affinities are evidently with the strophomenoids, especially such forms as Plectambonites, Leptænisca, etc. Briefly the reasons for this statement are (*a*) the presence of a deltidium of one plate; (*b*) the absence of a true loop supporting the arms (the internal calcification or spiculization is confined wholly to the mantle, and does not extend to the arms[16]); (*c*) a concave place in the cavity of the ventral beak, bearing the divaricator muscles; (*d*) the attached ventral valve, and (*e*) the cardinal processes in the dorsal valve.* The first character is of prime importance, because all the strophomenoids and none of the terebratuloids have a deltidium of one plate.

It would appear, therefore, that the early, free swimming, larval state, and the later pediculate stage have become lost by acceleration, thus accounting for the very unequal development of the mantle lobes in the cephalula stage, and the non-active and early sedentary larvæ as described by Kovalevski and Lacaze-Duthiers.

The young Lingula (Glottidia) described by Brooks,[4] and the Discinisca by Müller,[20] both representing the phylembryonic stage, were active and free swimming animals, with rudimentary pedicles. Terebratulina becomes attached or rests on the caudal segment during the cephalula stage (Morse), while at the end of this period in Cistella (Kovalevski and Shipley), there is an active, swimming, ciliated organism, which later attaches itself by the pedicle in the typembryonic period.

* Dall in 1870 (Am. Jour. Conchology) made a clear statement of the characters of Thecidium and of many of its radical points of difference with the Terebratulidæ, showing that it was entitled to rank as the type of a distinct family.

From the facts, that young individuals of paleozoic species belonging to such genera as Zygospira, Spirifer, Orthis, Rhynchonella, and Scenidium, have been observed by the writer to retain their original relations to the objects of support, and that casts of the pedicles of fossil Lingulæ and Eichwaldia have been described (Davidson,[6] Walcott[23]), it cannot be assumed that the free swimming condition was ever present in nealogic or ephebolic individuals. Evidently it has always been a larval character.

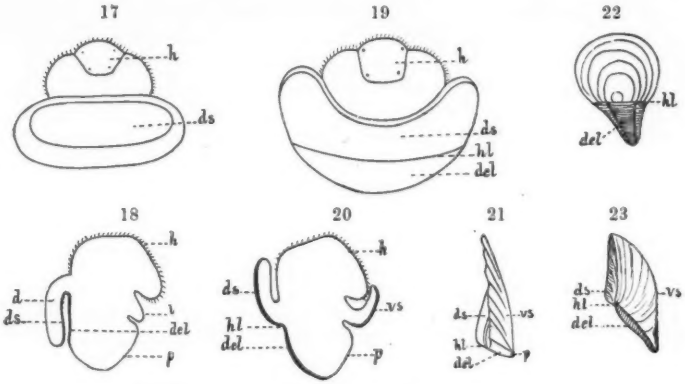
Origin of the deltidium and deltidial plates.

The origin and significance of the deltidium* (="pseudo-deltidium") are made apparent in the development of Thecidium, and it may be well in this place to make a few observations on the genesis of this important character, and its relations to the deltidial plates of other genera, as Rhynchonella and Terebratula. It has been already noted (Part I), that the deltidium in all species possessing it (the Protremata) is an embryological, or nepionic feature, which may or may not continue to the ephebolic period; while the deltidial plates in other brachiopods (the Telotremata) appear later during the nealogic and ephebolic periods, or may never be developed. The detailed researches of Kovalevski on Cistella and Thecidium, together with other observations now first made, furnish data for a clear understanding of these differences.†

Figure 18 represents a dorso-ventral section of a ripe cephalula just before the transformation, and shows the unequal lobes of the mantle, *v* being the ventral lobe, and *d* the dorsal; *h* is the head, and *p* the caudal segment developing into a pedicle. A deposit of integument representing the shell has formed on the inner side of the dorsal mantle lobe (*ds*), and also on the adjacent dorsal side of the body lobe (*del*). A larva somewhat more advanced is represented in figure 17, as viewed from the dorsal side. The mantle lobe is still directed posteriorly, as in the preceding figure, and the underlying shell plate is shown

* The single plate or covering to the triangular opening beneath the ventral beak should be termed the *deltidium*, as it was thus extensively used by Davidson. When it consists of two plates they may be called *deltidial plates*. These names have been loosely used. In Part I of this paper the deltidium proper is referred to as pedicle covering, pedicle sheath, and pseudo-deltidium. Hall and Clarke have proposed to call the triangular opening in the beaks of brachiopods, the *delthyrium*, and the concave plate in the ventral beak of Pentamerus, Orthisina, etc, they have termed the *spondylium*. There yet remains a term for the convex plate covering the opening below the beak of the dorsal valve, and resembling the deltidium of the opposite valve. For this feature the name *chilidium* (χειλος) is here proposed.

† Kovalevski.[15] For Thecidium consult the explanation of Pl. IV, figs. 15–26. For Cistella, Pl. I, figs. 13–15; Pl. II, figs. 17, 19–21.



Thecidium (*Lacazella*) *mediterraneum* Risso.

FIGURE 17.—Cephalula; dorsal side. *ds*, dorsal shell plate; *h*, head. (After Kovalevski.)

FIGURE 18.—Dorso-ventral longitudinal section of cephalula of about same age as preceding. *h*, head; *d*, dorsal mantle lobe; *v*, ventral mantle lobe; *ds*, beginning of dorsal valve; *del*, shell plate forming on dorsal side of body; *p*, pedicle. (After Kovalevski.)

FIGURE 19.—Typembryo. Larva transformed from folding upwards of mantle lobes. *h*, head; *ds*, dorsal valve; *hl*, hinge line of dorsal valve; *del*, shell plate on body and pedicle posterior to hinge line of dorsal valve. (After Kovalevski)

FIGURE 20.—Dorso-ventral longitudinal section of preceding. References as in figure 19. *vs*, ventral valve.

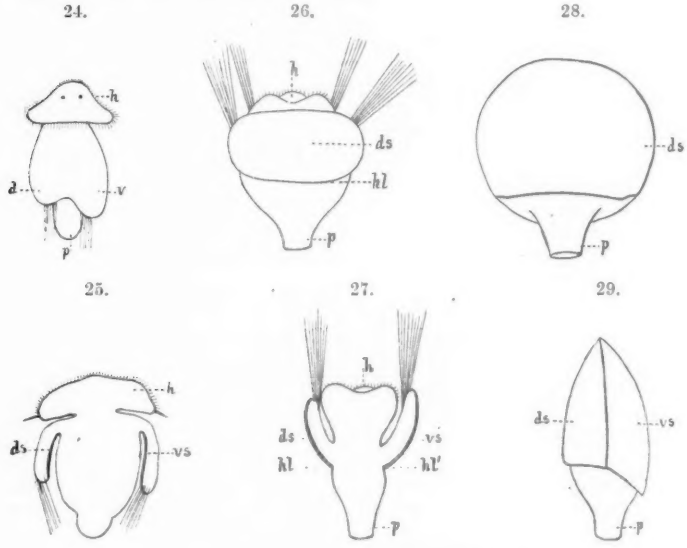
FIGURE 21.—Profile view of nealogic *Leptæna rhomboidalis*. The features of the shell are placed and lettered as in figure 20. *ds*, dorsal valve; *hl*, hinge line; *del*, deltidium; *p*, pedicle opening; *vs*, ventral valve.

FIGURE 22.—Adult *Thecidium* (*Lacazella*) *mediterraneum;* dorsal side; showing ventral area and deltidium.

FIGURE 23.—Profile of same. References as in figures 20 and 21.

at *ds*. In the process of transformation (figs. 19, 20), the mantle lobe is turned forwards in the usual manner, bringing the shell on the outside of the animal, so that both dorsal plates are now exposed, *ds* being the dorsal valve, and *del* the shell developed on the dorsal side of the walls of the body and caudal segments. As this plate (*del*) is below or posterior to the hinge line (*hl*), and extends down over the pedicle, it is evidently the beginning of the deltidium. At the same time, there is an extension of the edges of the mantle and pedicle on the ventral, or lower side, and shelly matter is deposited, forming the ventral valve (*vs*), figure 20. At this stage the hinge line, figures 19, 20, *hl*, is the line between the dorsal mantle shell (*ds*) and the dorsal body shell plate (*del*). The beak of the ventral valve is separated from the dorsal beak by the pedicle and the shell covering to the pedicle and body

lobe, or the deltidium. The valves afterwards meet at their peripheries, the hinge is extended beyond the deltidium, forming the true hinge of articulate brachiopods. As there is no motion between the ventral valve and deltidium, the two become ankylosed. Figures 22, 23, showing an adult Thecidium, are lettered in the same manner as the preceding, and express the same relation of parts.



Cistella neapolitana Scacchi.

FIGURE 24.—Lateral view of completed cephalula stage. *d*, dorsal lobe of mantle; *v*, ventral lobe. (After Shipley.)

FIGURE 25.—Dorso-ventral longitudinal section of same; showing posteriorly extended mantle lobes. *ds* and *vs*, inner surfaces of mantle lobes which are to form dorsal and ventral valves. (After Shipley.)

FIGURE 26.—Typembryo. Dorsal view of larva after transformation. *h*, head; *ds*, dorsal valve; *hl*, hinge line of dorsal valve; *p*, pedicle. (After Kovalevski.)

FIGURE 27.—Dorso-ventral longitudinal section based on preceding; showing mantle lobes directed forwards, bringing interior shell-secreting surfaces, *ds* and *vs* of figure 25, on the exterior. *h*, head; *ds*, dorsal valve; *hl*, dorsal hinge; *vs*, ventral valve; *hl'*, ventral hinge; *p*, pedicle.

FIGURE 28.—Dorsal view of early nepionic shell, showing large posterior opening between valves. (After Kovalevski.)

FIGURE 29.—Profile of same. *ds*, dorsal valve; *vs*, ventral valve; *p*, pedicle.

The deltidium is not, therefore, primarily, on account of its manner of origin, an integrant part of the ventral valve, but is a shell growth from the dorsal side of the body, which after-

wards becomes attached to the ventral valve, and is then considered as belonging to it.

The further growth of the deltidium around the body and pedicle, and its consequent extension into the cavity of the ventral umbo, may explain the origin of the spondylium.

Kovalevski[15] believed the ventral valve in Thecidium was secreted by the expanded edges of the pedicle and the body walls, whether or not this is so does not affect the interpretation of the origin of the deltidium. From the observations of Lacaze-Duthiers,[16] it seems, however, as though the ventral mantle lobe must have formed the shell in the usual way. This appears all the more probable from the fact, that the lower or ventral valve is punctate, and, so far as known, the mantle contains all the cæcal prolongations, which alone could produce the punctate structure. Careful microscopic examination has failed to detect punctæ in the deltidia of Thecidium, Strophomena, Leptæna, and other punctate genera belonging to the Protremata.

It is true that Aulosteges has spines on the deltidium, but spines even when tubular are not equivalent to punctæ, as shown in Productus, Strophalosia, and some species of Spirifer. Aulosteges is a geratalogous genus, which has become excessively spinose, and has also reverted to ancestral characters in its high hinge area and conspicuous deltidium. It is well known that even the spires of Spiriferina and the loop of Macandrevia are spinose.

Turning now to Cistella as a representative of the Telotremata, a different process obtains.

Figure 24 represents the fully developed, free swimming cephalula of Cistella, and shows the extent of the folds of the mantle and their posterior direction. Figure 25 represents the same in section. The inner sides of the mantle lobes are to form the future valves, the dorsal *ds*, and the ventral *vs*. The transformed larva or typembryo is represented in figure 26 and in section in figure 27. It is seen that the transformation consists in the folding forwards of the mantle lobes over the head segment *h*. Now the shell-secreting layers of the mantle are exterior, and the two valves begin to form, the dorsal shell *ds*, and the ventral *vs*. The pedicle and posterior portion of the body come out freely between the valves and mantle lobes and limit the hinge areas of both, *hl* anp *hl'*.

The further process of growth increases the distance between the initial dorsal and ventral hinges, for while the original dorsal beak is usually maintained at the hinge line, the ventral beak is progressively removed and the ventral hinge travels from its first position at the beak, along the edges of the umbo leaving an open triangular area or delthyrium in the

ventral valve occupied by the pedicle. This condition represents the extent of the development of these parts in *Meristina rectirostra* Hall or *Gwynia capsula* Jeffreys, which lack deltidial plates in the adult shell. The young of other telotremate species, as *Magellania flavescens* or *Terebratulina septentrionalis*, agree in the same respect.

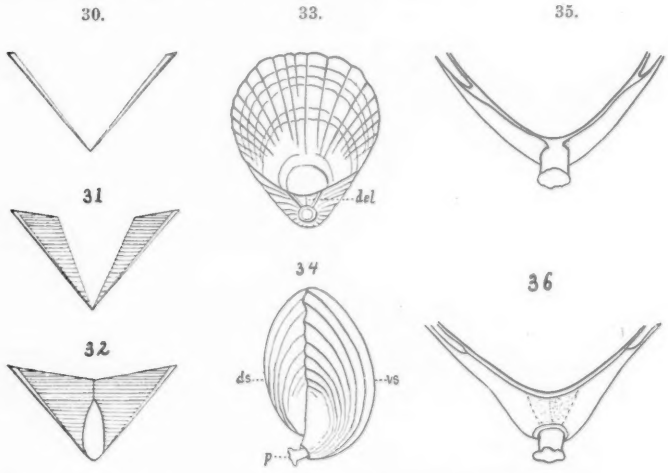


FIGURE 30.—Delthyrium of young Rhynchonella, without deltidial plates.
FIGURE 31.—The same at a later stage, with two triangular deltidial plates.
FIGURE 32.—The same after completed growth; showing joining of deltidial plates, and limitation of pedicle opening to ventral beak.
FIGURE 33.—Dorsal view of *Magellania flavescens;* showing completed deltidial plates, *del.*
FIGURE 34.—The same; profile. *ds*, dorsal valve; *vs*, ventral valve; *p*, pedicle.
FIGURE 35.—Dorsal view of umbonal portion of adult *Terebratulina septentrionalis*, with shell removed by acid; showing slight secondary extension of ventral mantle around pedicle (consequently small deltidial plates are secreted in this species). Mantle areas secreting deltidial plates are shaded.
FIGURE 36.—Dorsal view of umbonal portion of *Magellania flavescens*, with the shell removed by acid; showing the complete envelopment of base of pedicle by secondary expansions from ventral mantle, and consequent production of deltidial plates filling delthyrium except at pedicle opening. See figure 33.

An examination of the animal at this stage shows that the mantle lobes line only the interior of the valves proper. The exposed edges of the mantle are around the peripheries of the valves and also that portion of the ventral mantle border limiting the deltidial opening and passing along the sides of the pedicle at its base. The ventral mantle gradually extends from each side as two prolongations partially covering the opening and enveloping the proximal portion of the pedicle.

As this is an extension of the shell-secreting surface of the mantle, there naturally results the formation of two plates within the deltidial area. Their structure is commonly punctate whenever the valves are punctate.

These outgrowths or extensions of the mantle into the deltidial area finally touch and coalesce until, as in *M. flavescens*, the pedicle emerges through an opening in the ventral mantle, and *pari passu* the deltidial plates unite and limit the pedicle opening to the beak of the ventral valve. The latter process has been carefully described by Deslongchamps,[6] Clarke and the writer[5] and need not be dwelt on here. Figures 35 and 36 of the beaks of *T. septentrionalis* and *M. flavescens* with the shell removed show the relations of the ventral mantle to the pedicle, and the portions which secrete the deltidial plates.

The deltidium and delthyrium are often simulated in the growth of the *dorsal* valve in genera having a high cardinal area in this valve. Orthis, Leptæna, Clitambonites, Spirifer, and Stricklandinia, may be cited as examples. They cannot properly be correlated with similar parts in the ventral valve, for their origin is quite different. Primarily, a deltidial opening is for the extrusion of the pedicle and this belongs properly to the ventral valve. The dorsal fissure is the space between the diverging teeth sockets, and may be filled by the cardinal process, as in Leptæna and Orthis, or it may have in addition a convex plate or chilidium covering it, as in Clitambonites. In Spirifer and Stricklandinia, the opening remains unclosed.

The true deltidial plates are formed on the side of the pedicle adjacent to the hinge by extensions of the ventral mantle lobe, and begin as two plates. They are likewise expressive of maturity, and are of secondary development, while the deltidium begins as a single plate in the median line, and is eminently a primitive character in the Protremata.

From present knowledge of the group, it is difficult to offer an explanation for the presence of an anal opening in the Inarticulata and its absence in the recent Articulata, as the solution of the question depends upon whether the class is to be considered as progressive or degraded. The dorsal beaks of Amphigenia, Athyris, Cleiothyris, Atrypa, and Rhynchonella, are usually notched or perforate. The perforation comes from the union of the crural plates above the floor of the beak leaving a passage through to the apex. A similar opening occurs between the cardinal processes in Strophomena, Stropheodonta, and allied genera, and the chilidium may also be furrowed, as in *Leptæna* (=*Strophomena*) *rhomboidalis*. This character is evidently in no way connected with the pedicle opening,

but points to the existence, in the early articulate genera, of an anal opening dorsal to the axial line, as in the recent Crania. This dorsal foramen was described and figured by King[13] in 1850, Hall[8] in 1860 and by several authors since, and has commonly been termed a visceral foramen.

Œhlert[21] suggests that it was probably occupied by the terminal portion of the intestine. The persistence of the foramen seems to indicate an anal opening. In reference to this character and the obsolescence of the eyes the class must be viewed as retrogressive since paleozoic time. Other features, however, are manifestly progressive; namely, the gradual shortening, through time, of the posterior elements of the animal, as the pedicle, visceral portions, and internal shell structures, and the expansion of the anterior parts, as the shell and brachia.

A further advance in specialization is shown in the limitation of the pedicle opening wholly to the ventral valve in the higher rhynchonelloids, athyroids, spiriferoids, and terebratuloids. The absence of punctæ in all the early radicles and their subsequent development in the derived types may also have a similar bearing.

The features and importance of the protegulum have previously been discussed.[1] It is merely noticed here as the embryonic shell of the completed phylembryonic period, for it is the first stage which can be observed among the fossil species, and is the initial point for the discussions of the relations and affinities of recent and fossil forms. Of the protegulum and later stages, there is abundant material available in nearly every family of brachiopods, ranging through their entire geological history.

Post-embryonic stages.

In discussing the post-embryonic stages of growth two aspects of development must be clearly differentiated; (*a*) the ontogenetical, and (*b*) the phylogenetical. The ontogeny of a form like Schizocrania may be conveniently divided into the nepionic, nealogic, and ephebolic periods, and such stages may be clearly defined. The ephebolic stage of Schizocrania, however, is like a nealogic stage of Orbiculoidea. In other words, Orbiculoidea, in its development, passes through a Schizocrania-like stage before reaching maturity.* These facts must be viewed from a phylogenetic standpoint. Moreover, in the geological history of a group, certain ephebolic characters of

* Attention was called to this fact in a publication preliminary to vol. viii of the Palæontology of New York, pp. 131, 132, issued February, 1890. Also the development of the pedicle opening in Orbiculoidea was fully described.

early species may become accelerated, and pass into the nealogic period of later forms, while other characters remain ephebolic. Discinisca offers an illustration of this. Its nealogic characters agree with Orbiculoidea in the form of the valves and in the pedicle notch, but the circular or elliptical form of the dorsal valve in adult and nealogic Orbiculoidea appears so early in Discinisca, that it marks all the nepionic stages. The interpretation of these facts is, of course, very evident, and will be subsequently given in detail. Attention is here called to the statement, that while nepionic, nealogic, and ephebolic stages represent equal intervals in the life of each individual, they do not represent conditions of growth, or the possession of characters which always agree stage for stage in the species of one family or of different families.

Other distinctions to be made whenever possible are (*a*) whether certain characters (natural or acquired) belong to a species by inheritance, or (*b*) are mere adaptations to special conditions of environment arising at any time in its history. A clear understanding of the first will lead to the true phylogeny of a species or genus, but to reach this the characters of the second category must be excluded. Thus in the series of Schizocrania, Orbiculoidea, and Discinisca, already cited, there is an apparent genetic connection in the facts as stated. The contrary must be the case with a shell like *Lingula complanata* Williams, and *L. riciniformis* Hall which initiate a holoperipheral* mode of growth in the ephebolic period, for this agreement in the method of concrescence with adult Orbiculoidea here appears in the mature stages of this species, and being absent in the early members of the genus cannot therefore be an ancestral character. It is a morphological equivalent, which may or may not be continued in the later species of the series.

Whenever features are present which can be referred to an ancestral origin, their elimination can take place only by the process of acceleration of development. On the other hand, there may be secondary characters of dynamical or homoplastic origin which appear simultaneously or independently in different groups belonging to diverse genetic lines, as the deltidial plates of the Rhynchonellidæ, Terebratulidæ, and Spiriferidæ. Further, many such secondary features may occur anywhere in the geological history of the group, as the high hinge area of Orthisina, Spirifer, Syringothyris, and Thecidium. These statements are in full accord with what Hyatt has determined in the Cephalopoda, and the application of such ideas affords a fertile field of research.

* ὅλος, whole; περιφέρεια, circumference.

Preliminary to a study of the stages of growth observed in the different orders, a simple characteristic example of each will be taken to show the limitations of the post-embryonic periods.

Nepionic period.—In brachiopods, as in pelecypods, this period represents the growth of the true shell immediately succeeding the embryonic shell or protegulum, and before the appearance of definite specific characters. In general, the nepionic shells of all groups are marked only by fine concentric lines of growth, and are therefore nearly smooth. Sometimes, however, a few radiating striæ or other ornaments may appear over the nepionic portion, but this is not the prevailing rule. *Obolus pulcher* Matthew shows a cancellated nepionic stage and is one of the most striking exceptional examples.

Plate I, figure 1, represents the nepionic stage of *Glottidia albida*, drawn from the beak of a well-preserved adult. The shell at this period had a short straight hinge (originally the hinge of the protegulum), with lines representing anterior and lateral growth, making the outline broadly ovate. It is divided from the succeeding growth of later stages by a strong varix. The form is suggestive of Obolella, and as this is the early form of growth of many of the Lingulidæ and allied families, it is here called the *Obolella-stage*. It is not known that otherwise the characters agree with those of Obolella, but as it is characteristic as well as descriptive the name is used to designate this form of nepionic growth whenever present.

The nepionic stage of *Orbiculoidea minuta*, figure 4, shows a continuance of the straight hinged condition after the completion of the embryonic shell, with nearly equal incremental lines. As this agrees with the shell of Paterina it is called the *Paterina-stage*. The pedicle emerged freely between the cardinal margins of the valves. It will be shown that both this and the Obolella-stage are represented in the nepionic periods of many genera belonging to the Atremata. They may succeed each other in a single species or one alone may be present. In case both appear, the Paterina stage is always the first one to be developed.

The nepionic stage of *Leptæna* (=*Strophomena*) *rhomboidalis*, fig. 7, Pl. I, is represented by a shell without radii, having a comparatively large pedicle opening in the ventral valve and a large deltidium. The hinge is not well defined and the shell is discinoid in form. This term is not used to suggest any special affinities with true discinoid genera, as Orbiculoidea or Discinisca. The proper name for this stage is not yet apparent to the writer. The external characters as expressed by both valves are manifestly nearer to Kutorgina

than to any telotremate genus. Until the early forms belonging to the articulate brachiopods, especially to the orthoid and strophomenoid groups, have been thoroughly studied, the interpretation of the nepionic *Leptæna rhomboidalis* may be uncertain. It should be noted, however, that the young of Chonetes, Productus, Stropheodonta, Orthothetes, Leptæna, Plectambonites, and Strophomena, all have little or no indication of a straight hinge line, and that the extension of this member takes place during later nealogic and ephebolic growth. This in itself is significant, but is more marked when taken with the growth stages shown by some species of Strophomena which have after the protegulum, a Paterina-like stage, with straight hinge in dorsal valve, succeeded by holoperipheral, discinoid, nepionic growth, and finally a renewal of a straight hinged condition. Thus it has an early straight hinged form, which is lost during the next stage of growth, and again appears, and is progressively elongated during nealogic and ephebolic growth.

The nepionic stages of *Terebratulina septentrionalis*, fig. 10 Pl. I, represent a decreasing extension of the cardinal line from the protegulum, an open delthyrium, the absence of radii, and the introduction of the shell punctæ. The crura at this stage, as shown by Morse, are short and stout, and the loop is undeveloped.

Nealogic period.—During the progress of this period all the features which reach their complete growth in the adult organism are introduced and progressively developed. Usually they appear in succession, and gradually assume mature conditions. Thus in many species with radiate plications or striæ, a few radii appear in early nealogic growth, and are added to until the full number is present. Species with deltidial plates develop them in this period. The early stages may offer many points for comparison with the adult, but later stages usually differ little except in size. Figures 2, 5, 8, 11, Plate I, represent a nealogic stage in each of the four species taken as examples. Others from the same species could be given, but these suffice to show that one or more characteristic adult features have made their appearance.

Ephebolic period.—The period of complete normal growth, or the maximum of individual perfection. This corresponds to the adult, or mature organism, and is so well understood that no further explanation is necessary. For the sake of completing the series, the ephebolic shells of the species given are represented in figures 3, 6, 9, 12, Plate I.

Geratologic period.—The variations due to old age may be numerous and complex. As shown by Clarke and the writer,[3] the valves generally become thickened, and, as a consequence,

the margins are truncate or varicose, the vertical diameter of the shell is increased, the beaks involuted, and the margins of the valves often lose the ornamentation characteristic of the species. The deltidial plates or deltidium may be resorbed as well as the beaks of the valves. Usually the ephebolic characters disappear in inverse order to their introduction. This is called the *clinologic stage* of geratology by Hyatt. Thus in a normal adult brachiopod having a plicate shell and deltidial plates, which characters were introduced during the nealogic period, the expression of old age will be found in the absorption of the deltidial plates and in the obsolescence of the plications. Large specimens of *Terebratella transversa* Sow. often furnish examples of this clinologic stage.

The geratologic development of Bilobites[2] consists in the obsolescence, in *B. varicus* Con., of the bilobed form of the shell, thus reverting to an early nealogic condition equally characteristic of *B. bilobus* and *B. Verneuilianus*.

Another aspect of growth and decline is manifest when the size of individuals and the chronological history of groups are taken into consideration. Each genus and family began with small representatives, and rapidly developed the more radical varieties of structure. Then came the culmination and final reduction in size, with abundance of geratologous and pathologic forms. The oldest known shell with calcareous spires, Zygospira, is a comparatively minute form. Nearly all the types of the suborder to which this genus belongs (Helicopegmata) appear in the Upper Silurian. Species presenting the maximum size belong to the Devonian and Carboniferous. Before the extinction of the suborder in the Trias, the individuals are small, and such abnormal genera as Thecospira, Koninckina, and Amphiclina, abound. Productus begins with small species (Productella) in the Lower Devonian, and in the Carboniferous attains the largest dimensions of any known brachiopod (*P. giganteus*). During the Permian, the species have dwindled in size, and the geratologous Strophalosia and Aulosteges are the chief representatives.

The culmination of geratologous growth results in the reversion of the animal to its own nepionic period, and is called the *nostologic stage*. As this is an extreme condition, it can be found only in certain genera and species which have been developed by a process of accelerated geratologous heredity. If Gwynia* is accepted as a valid genus, it belongs to a pronounced nostologic type. The shell has a small internal plate on each side of the dorsal umbo, evidently the bases of crural

*Some authors have been disposed to consider this form as the young of a species not yet determined. It has also been referred to *Macandrevia cranium*, *Cistella cistellula*, and *C. neapolitana*. This question cannot at present be determined, although some characters of the shell indicate a mature organism.

plates. King,[14] the author of the genus, states that the labial appendages are attached directly to the shell, and not to a loop, as in other genera of the family. Cistella may be taken as a representative of nostologic development among the terebratuloids. The species are smooth, or pauciplicate, and small; deltidial plates obsolescent, loop more or less undeveloped. In *C. neapolitana*, the lamellæ of the loop are nearly obsolete and are free only near the crura, while the anterior portions are confluent with the valve (Shipley). A slight progression of these reversions would naturally result in a degenerate form like Gwynia, which is without a calcareous loop; with no surface ornamentation; deltidial plates absent, punctæ few and large, all of which features are strictly nepionic. Besides Cistella and Gwynia, other loop-bearing genera present nostologic features of importance in a natural classification. These consist mainly in their small size; the absence of surface ornaments; the obsolescence of deltidial plates, and the loss of a complete loop supporting the arms. In the Terebratulidæ, Kraussina and Platydia may be mentioned as belonging to geratologous types with a nostologic tendency. Likewise, in other groups, Atretia in the Rhynchonellidæ, and Strophalosia and Aulosteges in the Productidæ, are examples of nostologic types.

Cistella and Gwynia among the genera of brachiopods, therefore, bear the same relation to the terebratuloids that Baculites among the cephalopods bears to the ammonoids.

Synopsis.

Protembryo.—Ovum and segmented stages before formation of blastula cavity.

Mesembryo.—Blastosphere.

Metembryo.—Gastrula.

Neoembryo.—Trochosphere and cephalula, with posteriorly directed mantle lobes, and bundles of setæ from body segment.

Typembryo.—Larva with mantle lobes folded anteriorly over head segment.

Phylembryo.—Brachiopod covered by protegulum, tentacles of arms developed, bundles of setæ dehisced, definition of stomach and œsophagus, direct transformation of larval muscles into those corresponding to muscles of adult animal.

Deltidium.—A single plate developed at an early period by the body and pedicle of animal posterior to dorsal hinge, and later ankylosed to ventral valve.

Deltidial plates.—A nealogic and adult feature produced by the extensions of the ventral mantle lobe into the delthyrium.

Brachiopoda.—Retrogressive in loss of anal opening and eyes, progressive in concentration of posterior elements, expansion of anterior elements, and limitation of pedicle opening to one valve.

Nepionic period.—Young shells before the appearance of distinctive specific characters.

Nealogic period.—Progressive development of the specific features which reach their complete growth in the adult.

Ephebolic period.—Normal adult condition.

Geratologic period.—Special manifestations of old age in ontogeny and in phylogeny.

Nostologic types.—Extremes of geratology represented by Cistella, Gwynia, and Atretia.

Yale Museum, New Haven, Conn., May 31, 1892.

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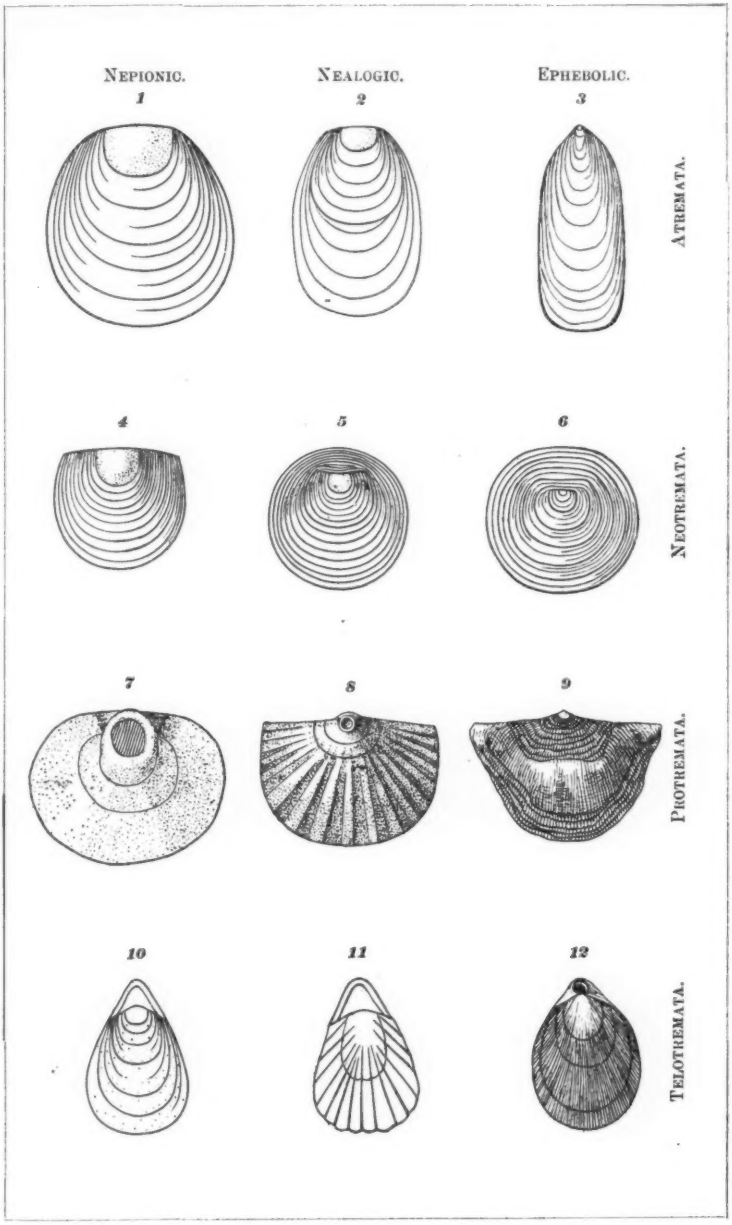

NEPIONIC.
NEALOGIC.
EPHEBOLIC.
1
2
3
ATREMATA.
4
5
6
NEOTREMATA.
7
8
9
PROTREMATA.
10
11
12
TELOTREMATA.

EXPLANATION OF PLATE.

Glottidia albida Hinds.

FIGURE 1.—Nepionic shell; Obolella stage. × 36.
FIGURE 2.—Nealogic stage; showing anterior growth producing Lingula-like form. × 16.
FIGURE 3.—Ephebolic stage. × $\frac{3}{4}$.

Orbiculoidea minuta Hall.

FIGURE 4.—Nepionic shell; Paterina stage. × 36.
FIGURE 5.—Nealogic stage; first holoperipheral growth. × 16.
FIGURE 6.—Ephebolic stage. × 10.

Leptæna rhomboidalis Wilck.

FIGURE 7.—Nepionic stages, with short hinge. × 36.
FIGURE 8.—Early nealogic stage, with radiating striæ. × 10.
FIGURE 9.—Ephebolic stage. × $\frac{3}{4}$.

Terebratulina septentrionalis Couth.

FIGURE 10.—Nepionic stage, with open delthyrium. × 26.
FIGURE 11.—Early nealogic stage, with radiating striae. × 16. (After Morse.)
FIGURE 12.—Ephebolic stage. × $\frac{3}{4}$. (After Davidson.)

ART. XX.—*On some Double Halides of Silver and the Alkali-metals;* by H. L. WELLS and H. L. WHEELER. *With their Crystallography;* by S. L. PENFIELD.

DURING a systematic search for well crystallized salts of the type M′Hl . AgHl,* which we were anxious to obtain on account of their probable isomorphism with the alkaline trihalides, three well defined compounds of another type, 2M′Hl . AgHl, were obtained. Our experience indicates that these 2 : 1 salts are more easily prepared and crystallize better than the 1 : 1 compounds.

The bodies to be described are 2CsCl . AgCl, 2RbI . AgI and 2KI . AgI. Two of these are believed to be new salts; the other, 2KI . AgI has been described by Boullay.† We have not obtained a complete series of these compounds, for good crystals could not be made of the other members, and, under the circumstances, no products were analyzed except such as could be measured.

The compounds are interesting from the fact that they do not conform to Remsen's law concerning the composition of double halides,‡ for, contrary to this, they contain a number of alkali-metal atoms which is greater than the number of halogen atoms belonging to the silver. In his latest contribution to the

* This Journal, III, xliii, 30 and 485. † Ann. Chim. Phys., II, xxiv, 377.
‡ Am. Chem. Jour., xi, 291.

subject,* Remsen states that the exceptions to his law are "not more than three or four out of over four hundred." The work here described confirms the result of Boullay, adds two more exceptions to the law and points to the existence of a greater number of compounds of the same type. It may be mentioned that a considerable number of other exceptions to this law have recently been established in this laboratory and will soon be described.

Preparation and properties.—The salts are made by saturating a very concentrated, hot solution of an alkaline halide with the corresponding silver halide, filtering, cooling to crystallization and, if necessary, evaporating the mother-liquor at ordinary temperatures. If the solutions are too dilute, in some cases at least, the 1 : 1 salts are formed. The compounds have little tendency to crystallize well and many trials are usually necessary in order to obtain satisfactory products. The salts are all white. They are readily decomposed by water.

Method of analysis.—The products analyzed were in the form of crystals of such size that it was certain that they were not mixed with other substances. In preparing them for analysis the mother liquor was removed rapidly and completely by pressing them between smooth filter-papers and great care was taken to avoid any evaporation of the liquid which adhered to them. The analyses were made by treating them with a sufficient amount of water acidified with nitric acid and weighing the silver halide thus separated. The filtrate from this was used for determining the remaining halogen or the alkali-metal.

ANALYSES.

	Found.	Calculated for 2CsCl . AgCl.
Cæsium	----	55·38
Silver	24·85	22·47
Chlorine	----	22·15

	Found.	Calculated for 2RbI . AgI.
Rubidium	25·05	25·91
Silver	17·32	16·36
Iodine	57·53	57·73
	99·90	100·00

	Found.	Calculated for 2KI . AgI.
Potassium	----	13·79
Silver	18·73	19·04
Iodine	----	67·17

* Am. Chem. Jour., xiv, 87.

Crystallography.—The three salts are isomorphous and crystallize in the orthorhombic system. The forms which were observed are:

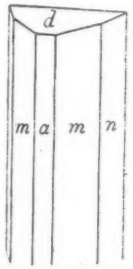
a, 100, *i-ī*	*m*, 110, *I*	*d*, 101, 1-*ī*
b, 010, *i-ĭ*	*n*, 120, *i-2̆*	*x*, 301, 3-*ī*

The axial ratios and some of the prominent angles are given in the following tables, the fundamental measurements being marked by an asterisk. The crystals did not yield very accurate measurements.

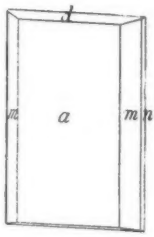
	$\breve{a} : \bar{b} : \dot{c}$
2CsCl . AgCl	0·971 : 1 : 0·244
2RbI . AgI	0·977 : 1 : 0·236
2KI . AgI	0·977 : 1 : 0·234

	$m \wedge m$, 110 $\wedge$ 110	$n \wedge n$, 120 $\wedge \bar{1}20$	$d \wedge d$, 101 $\wedge \bar{1}01$
2CsCl . AgCl	88° 18′	*54° 29′	*28′ 11′
2RbI . AgI	*88 40	54 12	*27 12
2KI . AgI	*88 40	54 12	*27 0

1.



2.



2 CsCl . AgCl was made in minute prisms, less than a millimeter in diameter, having the habit shown in fig. 1. The measurements are only approximately correct.

Two crops of 2RbI . AgI were examined. One was like fig. 1 in habit, the other in plates, fig. 2. The crystals were nearly 10^{mm} in length. On this salt a cleavage, parallel to *a*, was observed; also, as small faces, the forms *b* and *x*. which are not shown in the figures. In convergent polarized light an obtuse bisectrix was seen, normal to *a*, the axial plane being the brachy-pinacoid.

2KI . AgI was made in prismatic crystals, over 10^{mm} in length and having the habit and forms shown in fig. 1.

Sheffield Scientific School, April, 1892.

ART. XXI.—*On the Cæsium and Rubidium Chloraurates and Bromaurates;* by H. L. WELLS and H. L. WHEELER. *With their Crystallography;* by S. L. PENFIELD.

A STUDY of the compounds to be described was undertaken in the hope that some crystallographic analogy would exist between them and the alkaline pentahalides described in a previous article.* No such analogy has been found in spite of

*This Journal, xliv, 42.

the similarity of such formulæ as $CsCl.Cl_3I$ and $CsCl.Cl_3Au$, but since some of these gold salts have never been described and as they show some interesting relations among themselves, our results are deemed worthy of publication.*

Th. Rosenbladt,† in an article on the solubility of the chloraurates, states that the cæsium and rubidium salts lose their water of crystallization almost completely when dried over sulphuric acid. He gives no statement of the amount of water, but refers to his dissertation of 1872 which is inaccessible to us. He mentions, however, that the crystals of both salts belong to the monoclinic system, so that it is probable that the compounds he obtained were the ones that we have found to be anhydrous.

The compounds that have been prepared are $CsAuCl_4$, $2CsAuCl_4.H_2O$, $CsAuBr_4$, $RbAuCl_4$ and $RbAuBr_4$. We have attempted in each case to obtain bodies containing more cæsium and rubidium, but no evidence of their existence has been found.

An investigation of the corresponding iodine compounds was also undertaken, but, on account of the instability of auric iodide, we did not obtain any pure or well crystallized products.

Preparation:—The salts are so insoluble that they form precipitates when moderately concentrated solutions of the component salts are mixed, and the products are readily recrystallized from water or from the mother liquors. It is usually immaterial whether the solutions are neutral or acid or whether the gold or alkaline halide is in excess, but the salt $2CsAuCl_4.H_2O$ requires special conditions for its preparation, for it is apparently formed only when a large excess of gold chloride is present and when the solution does not contain much free acid. We have used four atoms of gold to one of cæsium in making this salt, but it usually requires repeated trials under these conditions before it is obtained free from the anhydrous compound. The two salts are however so distinct in form that there is no difficulty in distinguishing them.

Properties:—The color of $CsAuCl_4$ and of $2CsAuCl_4.H_2O$ is golden-yellow; $RbAuCl_4$ is yellowish-red; the two bromides are black, but give a dark red powder.

All the salts are sparingly soluble in water, especially when cold, and the cæsium compounds are less soluble than the rubidium. All of them are only slightly soluble in alcohol and insoluble in ether.

* The announcement by Professor Remsen (Am. Chem. Jour., xiv, 89), that he and Mr. H. C. Jones proposed to examine the gold-rubidium halides, was not made until after the work described in this article had been completed.

† Berichte, xix, 2535.

Methods of analysis:—The crystals were prepared for analysis by quickly pressing them between smooth filter-papers and finally allowing them to become air-dry. The hydrous cæsium chloraurate, however, loses its water and becomes opaque on exposure. It was therefore dried as rapidly and thoroughly as possible on paper and was put into a weighing-tube as soon as some of the fragments began to lose their transparency.

Gold was determined by precipitation with ammonium oxalate or with sulphurous acid. The filtrate from the metallic gold was used either to determine the alkali-metal as normal sulphate or the halogen by the usual gravimetric method. Water was determined by the method used in the combustion of organic compounds, the halogens being held back by a mixture of lead chromate and lead oxide. The absence of water in the anhydrous compounds was established by the use of the same process.

ANALYSES.

	Found.			Calculated for $CsAuCl_4$
Cæsium	28·11			28·16
Gold	41·61			41·77
Chlorine	29·91			30·06
	99·63			

	Found.			Calculated for $2CsAuCl_4 . H_2O$
Cæsium	27·23	----	----	27·63
Gold	40·23	----	----	40·99
Chlorine	29·07	----	----	29·50
Water	2·32	2·37*	2·20*	1·87
	98·86			

	Found.		Calculated for $CsAuBr_4$
Caesium	20·73	----	20·45
Gold	30·32	30·26	30·34
Bromine	49·31	----	49·21
	100·36		

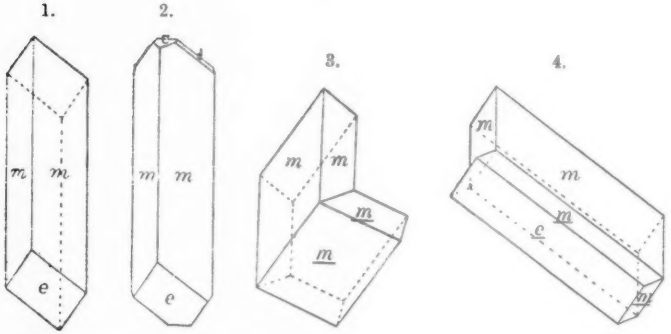
	Found.	Calculated for $RbAuCl_4$
Rubidium	----	20·14
Gold	45·53	46·46
Chlorine	32·98	33·40

* From a separate product.

	Found.	Calculated for $RbAuBr_4$
Rubidium -----	----	14·18
Gold ----------	32·54	32·73
Bromine ------	----	53·08

Crystallography:—The crystallization of $CsAuCl_4$, $CsAuBr_4$, $RbAuCl_4$ and $RbAuBr_4$ is monoclinic. The four salts form an isomorphous group and are identical in crystalline habit. The forms which have been observed on them are:

c, 001, O *d*, 021, 2-$\bar{i}$ *p*, $\bar{1}11$, 1
m, 110, I *e*, $\bar{2}01$, 2-$\bar{i}$



The crystals are prismatic and are usually terminated by *e*, fig. 1. When other faces are present they are always small, as represented in fig. 2. The pyramid *p*, which is not shown in the figure, frequently occurs as a small face, replacing the edge between *d* and *e*. Among the crystals of $CsAuBr_4$ several twins were observed, having *p*, $11\bar{1}$ as the twinning plane, fig. 3, while fig. 4 represents a crystal of $RbAuBr_4$ twinned about *e*, $20\bar{1}$. The letters belonging to the parts in twin position are underlined. Both kinds of twins are abnormally developed as represented in the figures. In all four compounds the cleavage is perfect parallel to the base.

The rubidium salts, being the most soluble, form readily in large crystals, several centimeters in length. The chloride, especially, yielded magnificent crystals, which frequently were only limited in length by the size of the vessel and volume of the solution containing them. The cæsium salts are less soluble and were made in small prisms, seldom over 5^{mm} in length. The crystals were frequently hollow or cavernous at the extremities, this was especially true of the two bromides. The faces, for the most part, gave excellent reflections of the signal on the goniometer.

The axial ratios are as follows:

$CsAuCl_4$	$CsAuBr_4$
$\dot{a}:\bar{b}:\dot{c} = 1{\cdot}1255:1:0{\cdot}7228$	$\dot{a}:\bar{b}:\dot{c} = 1{\cdot}1359:1:0{\cdot}7411$
$\beta = 71° 36'$	$\beta = 70° 24\frac{1}{2}'$
$RbAuCl_4$	$RbAuBr_4$
$\dot{a}:\bar{b}:\dot{c} = 1{\cdot}1954:1:0{\cdot}7385$	$\dot{a}:\bar{b}\ \dot{c}: = 1{\cdot}1951:1:0{\cdot}7256$
$\beta = 75° 32'$	$\beta = 76° 53\frac{1}{2}'$

In the following tables the angles which were chosen as fundamental are marked by an asterisk.

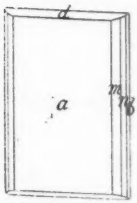
	$CsAuCl_4$			$CsAuBr_4$	
	Measured.	Calculated.		Measured.	Calculated.
$m \wedge m$, $110 \wedge 1\bar{1}0$ =	*93° 46′			*93° 53′	
$m \wedge c$, $110 \wedge 001$ =	77 36	77° 32′		*76 46	
$m \wedge d$, $110 \wedge 021$ =	44 6	44 7		43 23,	43° 20′
$d \wedge p$, $021 \wedge \bar{1}11$ =				32 23	32 40½
$d \wedge e$, $021 \wedge \bar{2}01$ =	*75 17			75 31	75 59
$m \wedge e$, $1\bar{1}0 \wedge \bar{2}01$ =	*60 36			*60 41	
$c \wedge e$, $001 \wedge \bar{2}01$ =	64 20,	64 18			
$m \wedge m$, Reëntrant angle of twin,				27 58	27 58

	$RbAuCl_4$			$RbAuBr_4$	
	Measured.	Calculated.		Measured.	Calculated.
$m \wedge m$, $110 \wedge 1\bar{1}0$ =	*98° 21′			*98° 40′	
$m \wedge c$, $110 \wedge 001$ =	*80 36			*81 30	
$m \wedge d$, $110 \wedge 021$ =				44 57	45 12½
$d \wedge p$, $021 \wedge \bar{1}11$ =				31 26	31 35½
$d \wedge e$, $021 \wedge \bar{2}01$ =				72 28	72 26
$m \wedge e$, $1\bar{1}0 \wedge \bar{2}01$ =	*62 12			62 9	62 21½
$c \wedge e$, $001 \wedge \bar{2}01$ =	60 4	59° 59′			
$d \wedge d$, $021 \wedge 0\bar{2}1$ =	110 20	110 4½		*109 26	
$m \wedge m$, Reëntrant angle of twin,				55 42	55 17

In their axial ratios the two cæsium salts are very similar, as are also the two rubidium salts, while the rubidium compounds differ considerably from those of cæsium, especially in the relation of $\dot{a}$ to the other axes and in the angles β. It is therefore evident that the replacement of one metal by another in these salts has a considerable influence upon their form, whereas, as we have shown, such a replacement in the cæsium and rubidium trihalides has little or no effect. There seems to be no regularity in the influence of the replacement of chlorine by bromine in these gold salts, for in the cæsium compounds the chloride has a slightly shorter axis $\dot{c}$ and a greater angle β than the bromide, while in the rubidium salts exactly the reverse is true in both cases. This unexpected relation between the chlorides and bromides has been confirmed by repeating the measurements, especially of the angle $m \wedge c$, using both crystal and cleavage faces. It is certain that this angle is about a degree greater with the chloride than with the bromide in

the cæsium salts, while in the rubidium compounds it is about a degree less.

5.



The crystallization of $2CsAuCl_4 . H_2O$ is orthorhombic. This salt was repeatedly made but only one crop of crystals was obtained which was suitable for measurement. These were thin plates, having the habit shown in fig. 5. They were not over 5^{mm} in length and were only a fraction of a millimeter thick. On removal from the mother liquor, or from a moist atmosphere, the transparent plates rapidly became opaque and the faces lost their luster so that only approximate measurements could be obtained.

The forms which were observed are:

a, 100, $\breve{i}$-$\bar{i}$	*m*, 110, I	*d*, 101, 1-$\bar{i}$
b, 010, $\bar{i}$-$\breve{i}$	*n*, 120, $\breve{i}$-$\breve{2}$	

The axial ratio is as follows:

$$\breve{a} : \bar{b} : \dot{c} = 0{\cdot}625 : 1 : 0{\cdot}24$$

The following measurements were made.

$a \wedge m$, $100 \wedge 110$ = about 32°	$a \wedge b$, $100 \wedge 010$ = about 90°
$a \wedge n$, $100 \wedge 120$ = " 51°	$d \wedge d$, $101 \wedge \bar{1}01$ = " 42°

Under the polarizing microscope the crystals show parallel extinction and, in convergent light, an acute bisectrix normal to *a*, 100. The plane of the optical axes is the base. The divergence of the axes is large, the hyperbolæ opening out beyond the field of the microscope. The axes of elasticity are:

$$\breve{a} = \mathfrak{c}, \qquad \bar{b} = \mathfrak{a}, \qquad \dot{c} = \mathfrak{b}.$$

The double refraction is therefore positive.

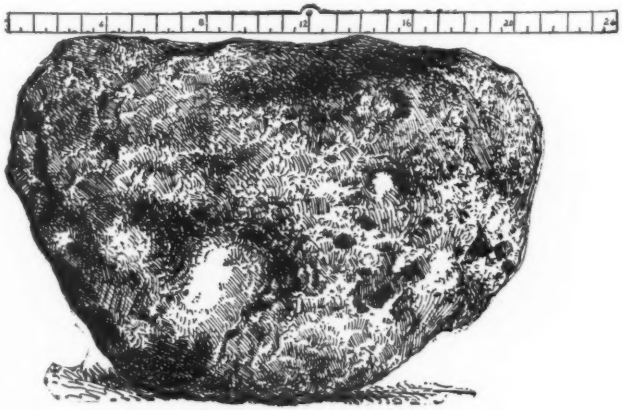
The change which the crystals undergo when exposed to dry air is a molecular rearrangement, accompanied by loss of water and, probably a change to the anhydrous salt which was described above. This rearrangement is a beautiful sight when studied with the microscope in polarized light. The change commences a few minutes after the crystals are removed from the mother liquor, and in less than ten minutes has usually advanced to such an extent that the crystals are no longer transparent. The crystals at first show a uniform action on polarized light, then from different parts of the surface the rearrangement, which is marked by aggregate polarization, commences. It advances, shooting out in various directions in a manner resembling the growth of ammonium chloride crystals under the microscope, until the whole field is covered and light is finally no longer transmitted.

Sheffield Scientific School, April, 1892.

ART. XXII.—*Preliminary Note of a New Meteorite from Kenton County, Kentucky;* by H. L. PRESTON.

[Read before the Rochester Academy of Science, June 27th, 1892.]

ON May 15th, Prof. Henry A. Ward received a letter from Mr. R. H. Fitzhugh, Bryson City, N. C., telling of a meteorite he had identified in Kenton County, Kentucky. In Prof. Ward's absence Mr. Frank A. Ward sent me off the same night to look up the meteorite. I arrived at Bracht station on the Cincinnati Southern R. R., Friday morning and drove as far as the roads would permit toward Mr. Geo. W. Cornelius's farm. He being away from home his wife showed me the "metal" as they called it. It proved to be a beautiful meteorite of the Siderite variety, 533×356×203 millimeters (21×14×8 inches) in its greatest diameters, and 163·0665 kilograms (359½ pounds) in weight.



Kenton Co. Meteorite, one-seventh natural size.

In form in certain directions it very much resembles a nautilus. It has numerous but mostly shallow pittings; a few deep pittings occur however on the side shown in the accompanying cut which gives a good idea of its general outline. This meteorite is entirely free from crust.

I saw Mr. Cornelius on the evening of the next day and obtained from him the following facts in relation to the meteorite.

About the middle of August, 1889, while cleaning out a spring situated at the head of a gully some three-quarters of a mile from his present home in Kenton County, eight miles south of Independence, the county seat, he struck with his hoe something that had a metallic ring; obtaining assistance he took the mass out, finding that it was interlocked in the roots of an ash tree from thirteen to fourteen inches in diameter and was between three and four feet below the normal surface.

He let the mass lie by the spring until August, 1890, when he removed it to his woodshed where it has lain until purchased by me for the Ward collection of meteorites. It is now at our establishment in Rochester, N. Y.

For the following analysis of this meteorite I am indebted to Mr. John M. Davison, of the Reynolds Laboratory, University of Rochester.

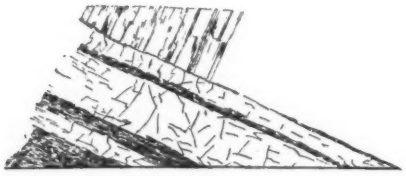
Fe	91·59
Ni	7·65
Co	0·84
Cu	trace.
C	0·12
S	trace.
P	trace.
	100·20

In the course of a conversation with Mr. S. J. Cornelius, a brother of the gentlemen of whom I purchased the meteorite, he mentioned the fact, that about three o'clock on the seventh of July, 1873, while returning from a picnic in this locality, and when within half a mile of where the meteorite was subsequently found, he heard a great rumbling in the heavens, which appeared to last three or four minutes and was followed by a quivering of the earth. As the day was clear he could not account for this phenomenon. I met at least seven other people who distinctly remembered the picnic and the "rumbling in the heavens," and some one or two "the quiver of the earth."

Is there any connection between this date and the fall of the meteor?

ART. XXIII.—*Additional observations on the Jura-Trias trap of the New Haven Region;* by JAMES D. DANA.

IN connection with my description of the south front of West Rock, the annexed figure of the eastern part of the exposed sandstone with a portion of the base of the overlying sheet of trap is introduced.* It is repeated here in order to mark from it the localities of two new observations of interest.



I. The exposure of sandstone represented in the above figure owes about eight feet of its height to the removal of overlying trap by the quarrymen. The trap quarry (see Plate VII of my former paper) extends from the point E eastward for nearly 140 yards. At a spot about 100 yards east of this point and 16 feet below it in level, the removal of the debris from the floor of the quarry has exposed the top of another ledge of upturned sandstone. The escape of the trap from the dike, therefore, was not along the sloping surface of the upper layer exposed at E, but along another about one hundred yards farther east.

II. About 90 feet west of the point E in the preceding figure, and 40 to 50 feet above the quarry-road, the talus with the partly concealed sandstone is crossed by a *five-inch dike of trap*. It was first observed last autumn by Professor W. O. Crosby. It looks from the quarry-road like a slight break in the steep surface. (On the phototype, Plate VII, referred to above, it is very faintly indicated across the bed of sandstone, just one inch from the sandstone point E.)

The little dike is a branch from the underside of the main sheet of trap. It makes a sinuous line of outcrops extending westward along the talus for about 40 feet, and then disappears under the debris. In this distance the dip of the outcrop is about 18 feet; but the true dip of the sheet is northwestward about 20° to 25°, as nearly as could be ascertained. Toward its junction with the main mass of the West Rock trap, it is reduced, for nearly a yard of its length, to two strands hardly

*This Journal, xlii, 102, 1891.

an inch thick by an intervening mass of sandstone; but this is only an irregularity of outflow due to the way the sandstone has of breaking, like that in the Mill Rock dike described and figured on page 88 of the paper above mentioned.

This branch dike has several points of interest.

1. It affords new proof that the West Rock outflow took place under a heavy cover of sandstone and therefore was laccolithic. This conclusion was sustained, in my former paper (p. 103), on the ground (1) that the outflow continued to be an ascending one for the 500 yards of its westward flow—which could not have been true unless it were under a resisting cover; and (2) the outflow retains a thickness of 250 feet quite to its extreme western limit, which it could not have done if it had been a subaerial, or, using a much needed new word, a *surficial*, flow.*

But (3) under *laccolithic* conditions, the opening of a fissure in the underlying sandstone for a discharge from the lower part of the mass of trap or else from the dike, would be a natural result. The liquid trap that was being forced up and onward under the repressing sandstone formation, might readily have made the fracture, especially when, after the lava had attained nearly its full thickness, a new start in its movement was given. Had it been a surficial stream it would have had no fracturing power.

2. Other points of interest in the dike are connected with its constitution. (1) While the rock of West Rock is a compact, rather coarsely crystalline doleryte, and has a light blue-gray color owing to the abundance of labradorite with the pyroxene, and is darker and finer but still gray at its junction with the sandstone, the trap of the dike is black, without luster, and aphanitic. Moreover, (2) it is amygdaloidal or vesicular; some of the cavities are over an inch in diameter and contain a lining of quartz crystals with a filling often of laumontite, while others are minute and filled with quartz. The rock is besides irregularly jointed, and much rifted and deeply altered along the rifts by weathering.

Examined microscopically in thin slices, the rock is found to have other peculiarities. (3) Magnetite is unusually abundant in very minute grains, much more so than in the trap of West Rock. (4) The labradorite of the rock is in crystals of the usual form, but they are extremely small. (5) The gray pyroxenic material about the labradorite is also in minute grains and seldom shows color in polarized light. Further, (6) *greenish olivine* is present in crystals and groups of crystals—a mineral not yet observed in the West Rock trap.†

* The word *superficial* is too various in its significations for the place. *Surficial* is like surface in having its prefix the French abbreviation *sur* in place of *super*.

† A full petrological description of the rock will be published later by another.

From the facts we are safe in concluding that the liquid rock of the little fissure had at its outflow the maximum temperature of the main mass; for it was from its lower portion. Evidence of high heat is indicated by the presence of olivine. For in the experiments of Fouqué and Michel Lévy, basalt on cooling after being for 48 hours at *white-red* fusion, "a temperature above the melting point of pyroxene and labradorite," afforded "crystals of olivine in a brownish vitreous magma;" but on cooling from *cherry-red* fusion sustained for 48 hours, afforded numerous microlites of labradorite and augite with magnetite.*

The olivine of the dike was made at the expense evidently of the pyroxene; and as ordinary olivine contains 8 to 10 per cent of iron protoxide and 41·5 of silica, and the pyroxene of West Rock trap, according to the analysis of Hawes,† 15·3 per cent of the former to 50·7 of the latter, some iron protoxide may have been set free in the process to add to the magnetite, besides silica to form the quartz-crystals and silicates in cavities or fissures.

The aphanitic texture of the trap indicates rapid cooling, and its vesicular character, cooling where there was much moisture. The temperature of the liquid trap was evidently too high to make chlorite from the constituents of the pyroxene. Moreover, the feldspar is not much altered notwithstanding the moisture at hand.

This dike, from the underside of the West Rock trap-mass, suggests an hypothesis with regard to the origin of the low and narrow belt of amygdaloidal trap that runs parallel with the high and wide belt of compact and nearly anhydrous trap of the Mt. Tom Ridge, from western Meriden northward, keeping in close parallelism with it and bending with it eastward at its southern extremity in the Meriden region. On Percival's map, Plate XVI in vol. xlii, of this Journal, the belt is that of the series of narrow dikes lettered A 1, situated just west of the areas 3, 4, 5, 6, 7, 8, 9, which mark the main trap range. It follows the curves and variations in height of the main range.

The hypothesis of Prof. Wm. M. Davis supposes that this low amygdaloidal belt is the outcrop of an inferior sheet of

* Synthèse des Minéraux et des Roches, 1882, p. 62.

† This Journal, III, ix, 187. G. W. Hawes obtained for the composition of the trap (doleryte) of West Rock (mean of two analyses): Silica 51·78, alumina 14·20, iron protoxide 8·25, iron sesquioxide 3·59, manganese protoxide 0·44, magnesia 7·63, lime 10·70, soda 2·14, potash 0·39, phosphoric acid 0·14, ignition 0·63=99·89; and for that of its pyroxene: Silica 50·71, alumina 3·55, iron protoxide 15·30, manganese protoxide 0·81, magnesia 13 63, lime 13·35, ignition 1·17, [alkalies and loss, 1·48]=100.

trap, parallel with that of the Mt. Tom Ridge; and that the two were raised together from their original horizontality along with the sandstone formation in which they were intercalated, into a monoclinal of 20° to 25°. The apparent satisfactoriness of this explanation is the strongest point in Mr. Davis's hypothesis.

The parallel courses of the two *unlike* belts has always seemed to me difficult to understand. But supposing the Mt. Tom ridge to be of laccolithic origin, like West Rock, the view I still believe to be most probable for reasons I have stated, the new facts from West Rock suggest an explanation: that this subordinate amygdaloidal belt of trap was produced by a lateral discharge from the dike of the laccolithic Mt. Tom belt, when the laccolithic discharge was nearing completion. This would account for the close relation of the two in position. Moreover the trap of such a dike would be sure to be amygdaloidal; for the north and south line of dikes of the Mt. Tom Ridge, situated along the western side of the Connecticut valley trough, would have been, from the commencement of the outflow, a barrier to the eastward or southeastward flow of subterranean waters descending from the north and west, so that the accumulated subterranean stream would have been large. To the eastward, the subterranean waters of the valley would have been divided up by the parallel trap dikes, for the hydration and vesiculation of other such branch dikes.

The value of the two hypotheses as to the origin of the trap belts—the dike or intrusive and the monocline—can be tested in two ways. One I have already referred to: the removal of the trap debris making the long east-and-west talus of the Mt. Tom ridge, near Meriden, exposing the sandstone, as done for the east-and-west talus along the similar south front of West Rock near New Haven. The sandstone may be, as it is at New Haven, *upturned* beneath the trap-mass, without conformability between the two rocks. If this were found to be the case, there would be no further ground for doubt as to the upturning before the outflow.

The other method is by boring. A boring carried down through the sandstone at points a short distance to the west of the Mt. Tom ridge would pass through, if the monocline hypothesis is the true one, at no great depth—probably within 1000 to 2000 feet of the surface—a layer of compact trap 200 to 300 feet thick, and then, after a little more sandstone, a layer of amygdaloidal trap; and not so, if the dike theory is correct. This test, repeated in other parts of the Connecticut valley in Connecticut and Massachusetts, would give conclusive facts.

The conformability of beds in monoclines is usually proved to be a fact by the study of *transverse* sections. But in the case of the trap and sandstone of the Jura-Trias area of Eastern America, no transverse section exhibiting such conformability between the trap and sandstone has yet been made known by any observer.

III. An emergence of the sandstone placing it more or less above the sea-level is necessary to render the method of hydration above appealed to possible. There is other evidence of such emergence.

The existence of dikes of amygdaloidal trap in East Haven, and of others of compact anhydrous trap in New Haven localities, two to five miles apart, is evidence, as explained in my Geology, of the hydration of the liquid rock in the former region while on the way to the surface; for the lava at its source must have been all alike. This must have taken place during its ascent through the sandstone; for, as shown by E. S. Dana, the trap of the related dikes that intersect the metamorphic rocks is unusually anhydrous. The New Haven dikes are situated along the western slope of the Connecticut valley trough, and those of East Haven toward or at its center, where the subterranean waters would have flowed. Hence the difference. But if the sandstone were wholly under the sea-level, the amount of subterranean water could not have thus differed, in the two regions; and consequently, there was some emergence.

If the sandstone formation was thus early emerged another conclusion follows. The trap of the Lake Saltonstall and Hartford Range (its more southern part marked EI, EII, EIII on Percival's map)—which I have shown to have originated, in all probability from fissure-ejections subsequently to the upturning of the sandstone—would have made, as I long since explained, a barrier to the Connecticut River waters, that would have cut off their flow southward and determined sooner or later their discharge by the present Middletown-Saybrook outlet. (See Percival's map.) These facts therefore bear on Prof. Davis's hypothesis, and on his deductions from it as to denudation over the region of the Connecticut valley.

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APPENDIX.

ART. XXIV. — *Notes on Mesozoic Vertebrate Fossils;* by O. C. MARSH. (With Plates II–V.)

THE extensive collections of vertebrate fossils from the Laramie now under investigation by the writer contain, besides those already described, many specimens of much interest, and some of these are briefly noticed and figured in the present article. The *Dinosauria* of this formation are of special importance, particularly in their relations to allied forms in the Jurassic, and hence some of the latter, also, are figured for comparison.

Claosaurus, Marsh, 1890.

Next in importance to the *Ceratopsidæ* of the Laramie are the Dinosaurs allied to *Hadrosaurus*, and, as but little is really known of the skeleton in this group, some of the important parts are here described, and figured in Plates II and III. These are mainly from a single specimen which is in remarkable preservation; but the remains of a second individual, likewise in good condition, and in some respects more perfect, have also been used in the investigation. The species is *Claosaurus annectens*, already briefly described by the writer.*

The skull will be described in a later communication. The number of vertebræ between the skull and sacrum is thirty, and all were found in position. There are nine vertebræ in the sacrum, thoroughly coössified with each other. The anterior forty-five vertebræ of the tail were found in position, and in good preservation.

* This Journal, vol. xliii, p. 453, May, 1892.

The fore limbs are unusually small in comparison with the posterior, and the relative size of the two is shown on Plate II. The scapular arch presents many points of interest. The scapula is large, and so much curved that the axis of its shaft is nearly parallel to the articular faces of its lower extremity, (Plate II, figure 1, *s*). On the anterior margin, above the articulation for the coracoid, is a strong protuberance, with a well-defined facet, adapted to the support of the clavicle, if such a bone were present. The coracoid is very small, and is perforated by a large foramen (Plate II, figure 1, *c*). The two peculiar bones now generally regarded as belonging to the sternum were separate, as shown in Plate III, figure 1.

The humerus is comparatively short, and has a prominent radial crest. The radius and ulna are much elongated, the latter being longer than the humerus, and the radius about the same length. The ulna has a prominent olecranon process, and is a stouter bone than the radius. The carpal bones were quite short, and appear to have been only imperfectly ossified. The fore foot, or manus, was very long, and contained three functional digits only. The first digit was rudimentary, the second and third were nearly equal in length, the fourth was shorter and less developed, and the fifth entirely wanting, as shown in Plate II, figure 1.

In the functional digits (II, III, IV), the phalanges are elongate, thus materially lengthening the fore foot. The terminal phalanges of these digits are broad and flat, showing that they were covered with hoofs, and not with claws. The limb as a whole was thus adapted to locomotion or support, and not at all for prehension, although this might have been expected from its small size and position.

The elongation of the fore-arm and manus is a peculiar feature, especially when taken in connection with the ungulate phalanges. It may, perhaps, be explained by supposing that the animal gradually assumed a more erect position until it became essentially a biped, while the fore limbs retained in a measure their primitive function, and did not become prehensile, which was the case in some allied forms.

The pelvis is shown in Plate II, figures 2 and 3, and has already been described by the writer. Its most notable features are seen in the pubis and ischium, the former having a very large expanded prepubis, with the postpubis rudimentary, while the shaft of the ischium is greatly elongated.

The femur is long, and the shaft nearly straight. The great trochanter is well developed, while the third trochanter is large and near the middle of the shaft, as shown in Plate II, figure 2. The external condyle of the distal end is projected well backward, indicating great freedom of motion at the knee.

The tibia is shorter than the femur, and has a prominent enemial crest. The distal end is much flattened, and the astragalus is closely adapted to it. The fibula is very straight, with its lower end flattened and closely applied to the front of the tibia. The calcaneum is large, with its concave upper surface closely fitted to the end of the fibula. Of the second row of tarsals, only a single one appears to be ossified, and that is very small and thin, and placed between the calcaneum and the fourth metatarsal, nearly or quite out of sight.

The hind foot, or pes, had but three digits, the second, third, and fourth, all well developed and massive. The terminal phalanges were covered with broad hoofs. The first and fifth digits were entirely wanting.

A comparison of the limbs and feet of *Claosaurus*, as here described and figured, with those of three allied forms from the Jurassic, *Stegosaurus*, *Laosaurus*, and *Camptosaurus*, as shown on Plates IV and V, is especially instructive. These three genera have already been quite fully described and figured by the writer, but new points of interest have been made out by the recent investigation of more perfect material. The present figures will show more accurately some of the mutual relations of these early herbivorous Dinosaurs to each other, as well as to their successors in Cretaceous time. The gradual changes that can be traced from one to the other will be discussed in a later communication.

Palæoscincus, Leidy, 1856.

A new reptilian genus and species, *Palæoscincus costatus*, was proposed by Dr. Leidy in 1856 for a single tooth found by Dr. Hayden in the Judith Basin. This tooth was more fully described and figured by Leidy in 1859.* The specimen showed well-marked characters, and many similar teeth have since been found, both in the Judith Basin and in various other localities of the Laramie.

A smaller species, apparently of the same genus, is not uncommon in the Ceratops beds of Wyoming, and a characteristic tooth is shown on Plate III, figure 3. This may be taken as the type specimen, and the species it represents may be called *Palæoscincus latus*. The crown of the tooth in this species is broader and the apex more pointed than in the first species described, and this is clearly shown in comparing the present figures on Plate III with those given by Leidy.

* Proc. Acad. Nat. Sci. Philadelphia, p. 72, 1856; and Trans. Amer. Phil. Soc., p. 146, pl. ix, figs. 49–52. 1859.

The tooth from the Laramie described by Cope in 1882 as a mammalian premolar and as the type of the generic name *Meniscoëssus* evidently belongs to the above or an allied genus, and all three are unquestionably the teeth of Dinosaurian reptiles pertaining to the order *Stegosauria*. On Plate IV, figure 1, a very small but typical tooth of *Stegosaurus* from the Jurassic is represented. The allied genus *Diracodon*, also Jurassic, has similar teeth.

Aublysodon, Leidy, 1868.

In the same publications above cited, Dr. Leidy also described and figured, under the name *Deinodon*, a number of teeth which he regarded as pertaining to carnivorous Dinosaurs, but later, in 1868, he made a new genus, *Aublysodon*, for some of these teeth which differed materially in form from those known to belong to such Dinosaurs.* The teeth regarded by Leidy as characteristic of *Aublysodon* are represented in figures 35–45, Plate IX, of the Transactions above quoted, and the best preserved tooth of this series, which Leidy suspected to be an incisor, is shown in figures 41–45. The latter figures are carefully reproduced on Plate III, figure 4, of the present article, and two other similar teeth are represented on the same plate. They all have the same characteristic chisel-shaped crowns, covered with a thin coat of enamel, and show indications of wear.

The teeth referred by Leidy to the genus *Aublysodon* and many others of the same general character since discovered may be divided into the four following groups, all the specimens of which appear to be somewhat curved either to the right or left:

(1) Large teeth (Leidy's figures 37–40) having both edges crenulated, and the posterior ridge between them broad. The wear of the apex is apparently posterior.

(2) Somewhat smaller teeth, but still large, one of which is represented in Plate III, figure 5. Faint crenulations may be detected on the edges. The wear of the apex is on front and back, and also on the side, probably the outside. The posterior central ridge is narrow. This tooth represents a distinct species which may be called *Aublysodon amplus*.

(3) Smaller teeth with no crenulations, and the posterior ridge with a groove (Leidy's figures 41–45). The wear of the apex is in front. These may be regarded as typical of *Aublysodon mirandus*, Leidy.

(4) The most abundant teeth are much smaller, with no crenulations, and the posterior ridge sharp and not grooved.

* Proc. Acad. Nat. Sci. Philadelphia, p. 198, 1868.

The wear is in front of the apex, and on one side, sometimes on both sides, as in figure 6, Plate III. This tooth may be taken as the type of a new species, *Aublysodon cristatus*.

The fact that these peculiar teeth are apparently in pairs, and are in themselves more like the teeth of mammals than of reptiles, has long been considered by the writer an argument for the mammalian character of the smaller forms at least. The large crenulated teeth described by Leidy strongly resemble those of carnivorous Dinosaurs, as he considered them, but no Dinosaur teeth of this form have been found in position in the jaws. The next smaller size, with very faint crenulations, one of which is figured in Plate III, figure 5, is too large for any mammal yet known from the Laramie, and this is true, also, of those figured by Leidy.

Many of the smaller teeth of this type, if considered apart from the others, would naturally be regarded as mammalian incisors, especially from the lower jaw, and the wear of the summits would in itself tend to strengthen this reference, if some of these teeth alone were considered. A number have been found, however, that show wear not only on the summit and on one side near the summit, but also on the other edge. This would imply, if these teeth are really lower incisors, either that the rami of the lower jaw were so loosely united at the symphysis that motion between them was possible, so that the incisors could thus rub against each other, or that these teeth were separated so as to admit the upper opposing teeth between them.

That some of these teeth are mammalian incisors there can be but little doubt, and this doubt can only be removed entirely by the fortunate discovery of a tooth in position in the jaw.

Cimolopteryx, Marsh, 1889.

The only bird hitherto known from the Laramie deposits is *Cimolopteryx rarus*, the type specimen of which is represented on Plate III, figure 2. Another species, about twice the size of the first, is indicated by various remains, among them the coracoid. This bone lacks the strong inner process near the pit for the scapula, which is characteristic of the smaller form. The present species, which may be called *Cimolopteryx retusus*, is also from Wyoming.

The new Laramie fossils here described and figured were collected by Mr. J. B. Hatcher and party, in the Ceratops beds of Montana and Wyoming. They will all be discussed more fully in another communication.

New Haven, Conn., July 18, 1892.

Explanation of Plates.

Plate II.

Figure 1.—Left fore leg of *Claosaurus annectens*, Marsh; outside view. *c*, coracoid; *h*, humerus; *r*, radius; *s*, scapula; *u*, ulna; I. first digit; IV. fourth digit.

Figure 2.—Left hind leg of the same individual; outside view. *a*, astragalus; *c*, calcaneum; *f*, femur; *f'*, fibula; *il*, ilium; *is*, ischium; *p*, pubis; *p'*, postpubis; *t*, tibia. Figures 1 and 2 are one-twentieth natural size.

Figure 3.—Pelvis of the same individual; seen from the left. One-sixteenth natural size. *a*, acetabulum; other letters as in figure 2.

Plate III.

Figure 1.—Sternal bone of *Claosaurus annectens*. One-eighth natural size. *a*, seen from above; *b*, seen from below.

Figure 2.—Left coracoid of *Cimolopteryx rarus*, Marsh. Natural size. *a*, front view; *b*, inner view; *c*, back view; *d*, lower end.

Figure 3.—Tooth of *Palæoscincus latus*, Marsh. *a*, natural size; *b*, *c*, *d*, twice natural size.

Figure 4.—Tooth of *Aublysodon mirandus*, Leidy. Natural size. *a*, front view, with sections; *b*, side view. (After Leidy.)

Figure 5.—Tooth of *Aublysodon amplus*, Marsh. Natural size. *a*, side view; *b*, back view; *c*, front view.

Figure 6.—Tooth of *Aublysodon cristatus*, Marsh. Twice natural size. *a*, side view; *b*, back view; *c*, front view.

Plate IV.

Figure 1.—Tooth of *Stegosaurus ungulatus*, Marsh. *a*, natural size; *b*, *c*, *d*, twice natural size.

Figure 2.—Left fore leg of the same species.

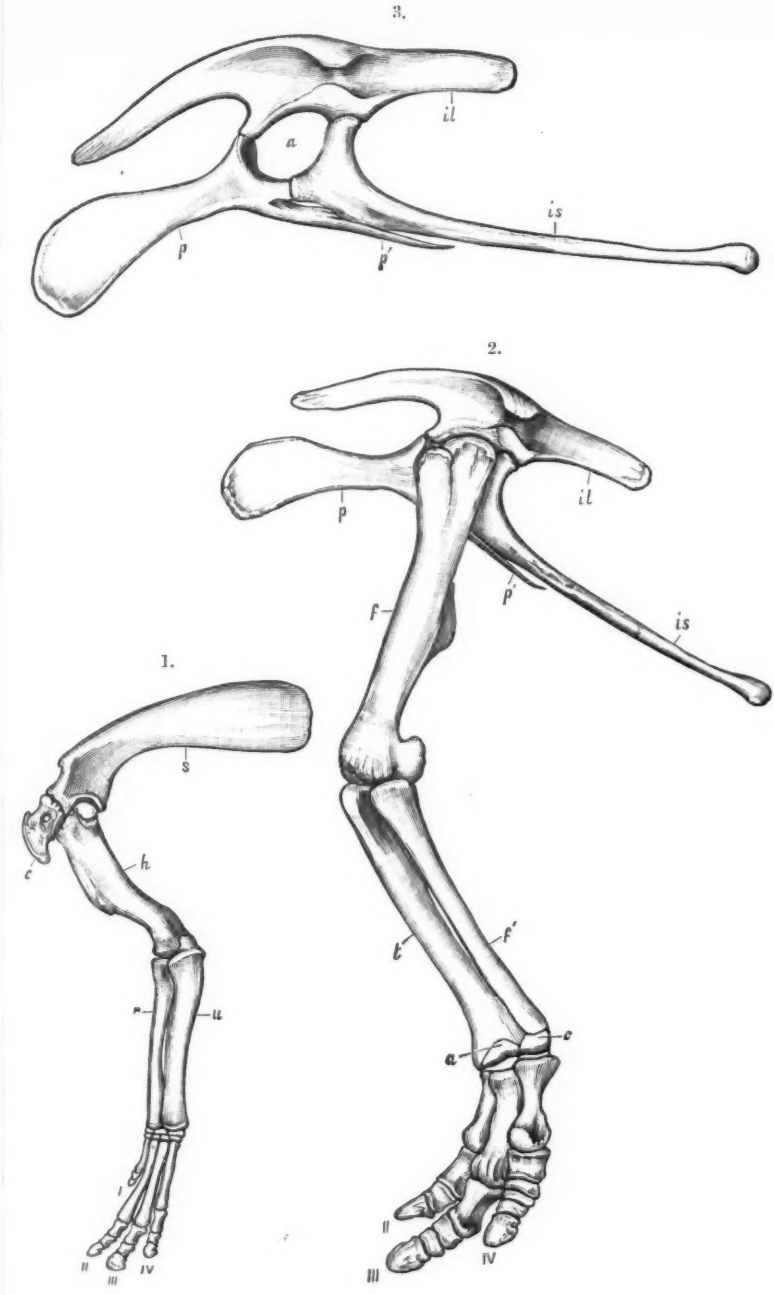
Figure 3.—Left hind leg of the same species. Figures 2 and 3 are one-sixteenth natural size. Letters as in Plate II.

Plate V.

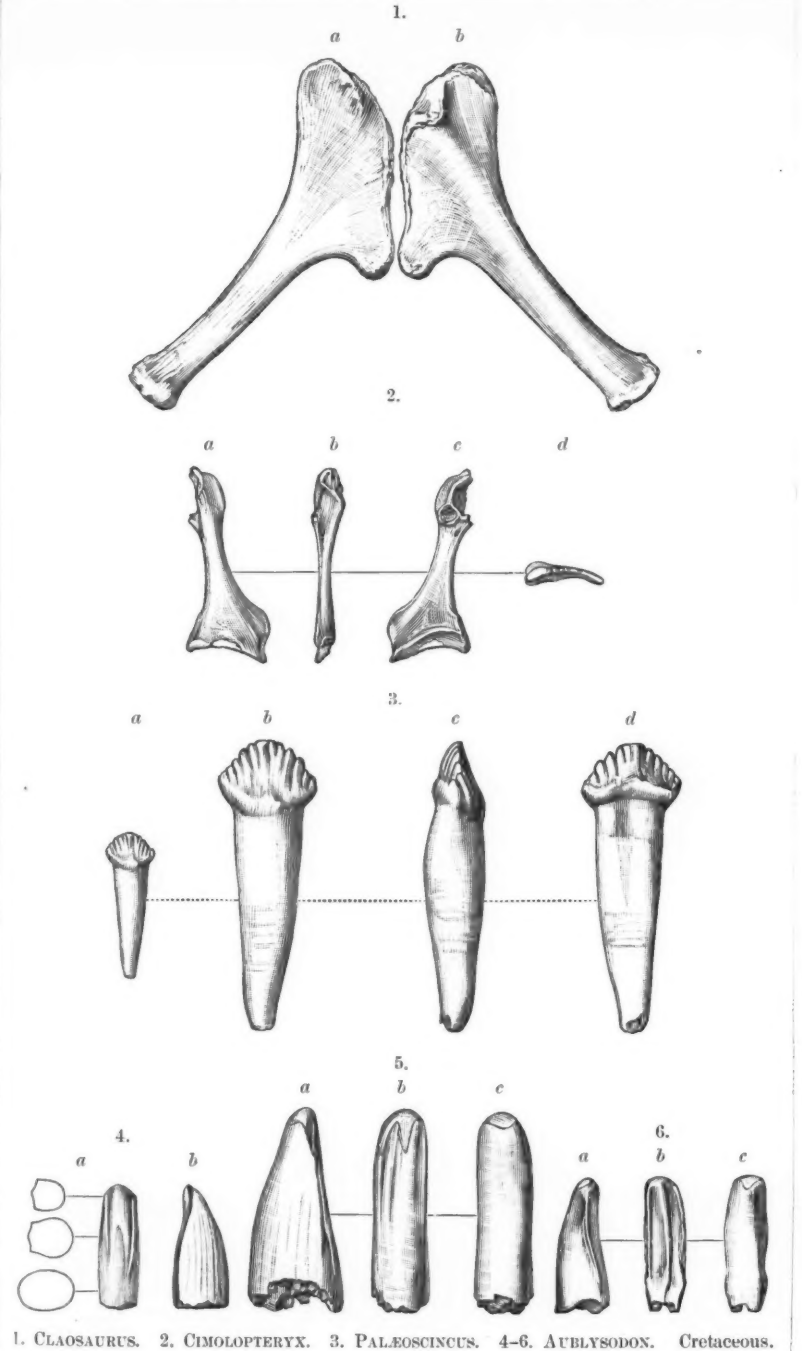
Figure 1.—Left hind leg of *Laosaurus altus*, Marsh; outside view. One-eighth natural size.

Figure 2.—Left hind leg of *Camptosaurus dispar*, Marsh; outside view. One-twelfth natural size.

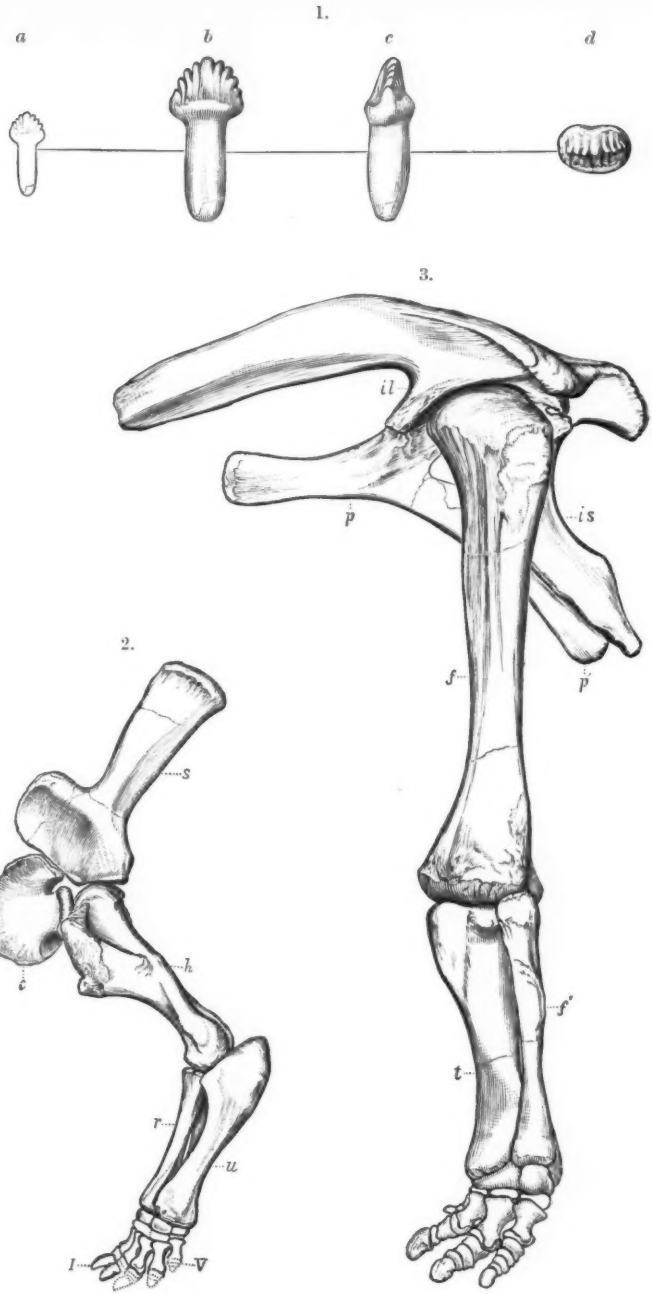
Figure 3.—Pelvis of the same individual; seen from the left. One-twelfth natural size. Letters as in the preceding plates.



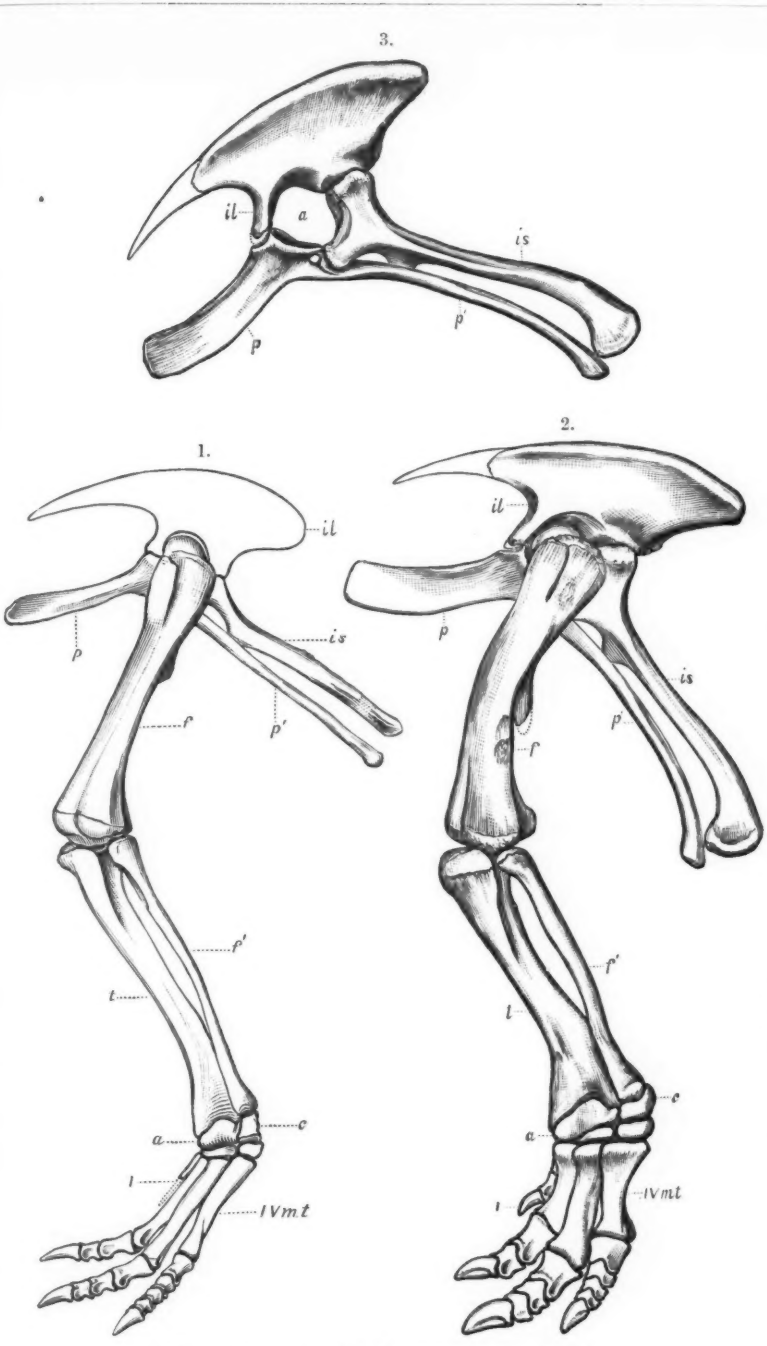
CLAOSAURUS ANNECTENS, Marsh. Cretaceous.



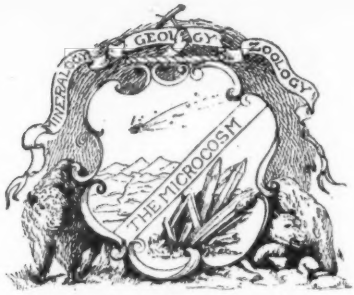
1. Claosaurus. 2. Cimolopteryx. 3. Palæoscincus. 4–6. Aublysodon. Cretaceous.



STEGOSAURUS UNGULATUS, Marsh. Jurassic.



1. Laosaurus. 2 and 3. Camptosaurus. Jurassic.



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